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BUTANE STRIPPING OF ORGANIC CONTAMINANTS FROM WATER - PHASE I

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BUTANE STRIPPING OF ORGANIC CONTAMINANTS FROM WATER - PHASE I

by

Pan Canadian Consultants Limited
North Vancouver, British Columbia

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1. EXECUTIVE SUMMARY

Pan Canadian Consultants Limited submitted an Unsolicited Proposal to the Federal government concerning a novel method of removing polluting organic chemicals from water. The proposal was accepted for funding by Supply and Services Canada and Environment Canada, and Pan Canadian Consultants Limited was awarded a contract to investigate the proposed process.

A laboratory test apparatus was designed and developed, and a test program was completed on a range of Priority Pollutants specified by Environment Canada. The test program entailed liquid-liquid extraction of the specified pollutants from water using liquid n-butane at 20°C and about 240 kPa. The process is referred to as "Butane stripping".

Results from the tests showed that of the 9 chemicals tested, 6 could be almost completely removed from water (97.5-99.8% removal), 2 could be partially removed (20-25% removal) and only one (phenol) could not be removed practically via butane stripping. These results were obtained in a simple, single-stage unit in the laboratory. A carefully engineered multi-stage unit should allow 99.99-99.9999% removal of certain organic pollutants from water.

Removal of 95-98% of the dissolved butane from the water was demonstrated in a simple laboratory experiment. We project the recovery of 99% of the butane in a suitably designed water de-butanizer, and its return to the process for re-use. The remaining dissolved butane would be air stripped from the water and lost to the atmosphere.

The mixture of butane and organic pollutant (after the butane stripping) can be separated in a fractionation column. Computer simulation shows that the butane can be fractionated as a high purity overhead product (99.999% butane) suitable for recycling to the process, with about 1% leaving as part of the bottom product. The butane in the bottom product is recoverable by distillation, and the extracted organic pollutant can be concentrated for subsequent recovery or incineration.

Butane stripping has been shown to be a technically feasible process for removing organic pollutants from water. Further, it is predicted to be an economical process, requiring relatively low energy input. Estimated energy costs to treat 1,000 litres of water are $1.13 (average) to $1.53 (worst). We believe this compares favourably with other processes.

Résumé

La proposition spontanée de Pan Canadian Consultants Limited portant sur l'évaluation d'une nouvelle technique de traitement d'eau contaminée par des produits chimiques organiques, extraction par butanol liquide, fût subventionnée par Approvisionnement et Service Canada et Environnement Canada.

Un contacteur de type laboratoire fût développé et utilisé avec des contaminants choisis parmi la liste des polluants prioritaires d'Environnement Canada. Une extraction de type liquide-liquide a été faite sur ces polluants avec du n-butane liquide à 20°C et environ 240 kPa. Le processus est ici appelé "strippage au butane".

Les résultats de ces tests ont montré que parmi les neuf produits chimiques traités, six d'entre eux peuvent être presque complètement séparés de la phase aqueuse (97.5 - 99.8% d'efficacité), deux peuvent être partiellement séparés (20 - 25% d'efficacité) et seulement un (phénol) n'a pu être à toute fin pratique, séparé par un strippage au butane. Ces résultats ayant été obtenus avec un simple contacteur à une étape, une efficacité d'enlèvement de 99.99 - 99.9999% devrait être possible avec un contacteur plus élaboré et à étapes multiples.

L'enlèvement de 95-98% du butane dissout dans l'eau a été possible avec un seul traitement primaire. On estime pouvoir récupérer 99% du butane avec un débutaniseur conventionnel et pouvoir le réutiliser dans le procédé. Le butane dissout restant peut être enlevé par strippage à l'air et évacué dans l'atmosphère.

Le mélange, butane et contaminants organiques, obtenu par le strippage au butane peut être séparé dans une colonne de fractionnement. Une simulation sur ordinateur a montré que le butane peut être séparé avec une pureté de 99.999% à la partie supérieure de la colonne. Il peut ainsi être recyclé dans le procédé. Environ 1% serait perdu dans les produits qui se retrouvent au bas de la colonne. Cette portion est récupérable par distillation et les contaminants organiques extraits peuvent être concentrés à nouveau par une autre technique pour ensuite être récupérés ou incinérés.

Cette étude a démontré que le strippage au butane est techniquement réalisable pour séparer les produits organiques chimiques d'une eau contaminée. On estime que le procédé est relativement économique, nécessitant très peu d'énergie pour son opération. Les coûts d'énergie pour traiter 1,000 litres d'eau contaminée sont estimés à $1.13 - $1.53 (moyen-pire). Ils sembleraient comparables aux coûts de traitements alternatifs.

2. INTRODUCTION

This project arose from a request from Environment Canada for information from companies interested in participating in a project to utilize steam stripping as an environmental emergency countermeasure. The proposed steam stripping project was directed towards the removal of volatile organic chemicals (for example, ethylene dichloride) from groundwater sources. The chemicals can enter groundwater via spills, leaking chemical storage tanks, etc. Most of the chemicals of concern are toxic.

On reviewing the request for information, Pan Canadian Consultants Limited (PCCL) felt that a superior alternative process was possible and should be investigated. The process proposed by PCCL was the solvent stripping of water using a volatile solvent that was immiscible with water yet would be an effective solvent for organics, and that had very low toxicity. The solvent proposed was n-butane.

PCCL believed that the proposed butane stripping process would have advantages over steam stripping in the areas of:

- lower energy requirements;
- higher extraction efficiency;
- more flexible operation; and
- easier waste disposal and handling.

As a consequence of PCCL's internal evaluation, a formal proposal was prepared and submitted to the Federal Government under the Unsolicited Proposals Program. The proposal was accepted for funding by Environment Canada and by Supply and Services Canada and a contract was awarded in July, 1987. With the funding from the contract, a program of theoretical and empirical investigation was initiated by PCCL in conjunction with faculty members at the University of British Columbia.

The project work undertaken, both in the office and in the laboratory, is described in this report.

3. PROJECT OBJECTIVES

The **main** objective of the project was to determine if butane could be used successfully as a solvent to strip contaminating organic chemicals from water, and to compare its effectiveness with steam stripping.

The **secondary** objectives of the project were to:

- perform an extensive literature search for data on: butane properties; relative solubilities of butane and water; whether any similar solvent stripping process has been researched or applied previously; solubilities of organic contaminants in butane; azeotrope formation; etc.

- derive a preliminary mathematical model for the process.

- calculate the energy requirements, and energy and mass balances for the process.

- develop, if possible, a flow sheet for a practical pilot-plant that might be developed to further examine the process if it appears likely to be a success from the initial project work.

4. DESCRIPTION OF THE TEST PROGRAM

4.1 Literature review

4.1.1 Summary

The literature review indicated that the process may be feasible, and that a limited amount of similar type work (using i-butane and i-butylene) had been done by the Environmental Protection Agency (EPA) in the U.S. In brief, the literature search showed:

- solvent extraction is a common process in industry although it is used infrequently as a waste water treatment process;
- the most common waste water treatment applications are for components whose recovery is considered to be of economic value (eg. PCB's, acetic acid, etc.);
- two feasibility studies were conducted by the EPA to investigate the use of volatile solvents for organics recovery. As was felt by PCCL staff in conceiving this project, the EPA felt that using a volatile solvent would be cost effective, since solvent recovery would have low energy requirements. One of these studies was aimed at finding an "all-purpose" solvent for waste water treatment applications where components are varied. Iso-butane and i-butylene were evaluated with a number of organic contaminants.
- the above EPA studies concluded that volatile solvent waste water treatment processes are promising and competitive with steam stripping. It was noted, however, that phenol was not extracted effectively from water using i-butane.

4.1.2 Theoretical basis for solvent extraction

Solvent extraction is a commonly used chemical engineering process. It's application is described succinctly by Perry's Chemical Engineers Handbook (Sixth Ed.):

> "The separation of components by liquid-liquid extraction depends primarily on the thermodynamic distribution of those components between the two liquid phases. Knowledge of these distribution

> relationships is essential for selecting the ratio of solvent to feed that enters an extraction process and for evaluating the mass-transfer rates or theoretical stage efficiencies achieved in process equipment. Since two liquid phases that are immiscible are used, the thermodynamic equilibrium involves considerable evaluation of non-ideal solutions. In the simplest case a feed solvent F contains a solute that is to be transferred into an extraction solvent S." (p.15-5)

In liquid-liquid extraction, a solute is distributed between two immiscible phases - the extract phase (E) and the raffinate phase (R). Upon mixing, the solute distribution moves towards equilibrium, at which point:

$$K_D = W_E / W_R$$

Where K_D is the equilibrium distribution coefficient, and W_E and W_R are the weight fractions of the solute in the extract (organic) and raffinate (water) phases (Ref. Barbarl et al, 1982)

For sparingly soluble compounds, the activity coefficient in the aqueous phase (τ_R) is the reciprocal of the mole-fraction solubility in water. Therefore K_D can be expressed as:

$$K_D = \tau_E/\tau_R$$

Where τ_E is the activity coefficient of the solute in the organic phase. Therefore, if values for activity coefficient are known or can be calculated, it should be possible to calculate values for K_D and vice versa.

Earhart et al (1976), and other researchers, describe procedures for calculating activity coefficients for many organics in aqueous solutions, and for predicting distribution coefficients via the UNIFAC model.

References in the literature (e.g., Won and Prausnitz, 1974) describe the experimental results obtained from tests using a variety of solutes and extractants in aqueous systems. From these the K_D values reported are high enough to make solvent extraction appear to be very promising compared with steam stripping, especially when the solute is a non-polar organic.

4.1.3 General considerations of solvent extraction

Liquid-liquid extraction has several potential attractions for the removal of contaminants from water (Humphrey et al, 1984):

1. There is the possibility of energy savings compared with distillation;

2. There is the possibility of recovering the contaminant as a marketable or useful product;

3. Effective contacting devices have been developed; and

4. The principles of extraction processes are better understood and scale-up of bench-scale processes is now more predictable.

Selection of process technology to be used for liquid-liquid extraction depends largely on the desired results. In the case of contaminated water, the desired result is to remove the contamination and to return the water to its uncontaminated state, and to do so in a process that is transportable and economic. Key factors that apply in this specific case are as follows.

Solvent selection

Key Factors in selecting a solvent for removal of organic contaminants from water, and properties such a solvent should have, are:

- low toxicity
- relative insolubility in water
- high selectivity for the contaminant(s)
- universality (as much as possible)
- easily obtainable and inexpensive
- easily recoverable
- easily separable from water.

From the initial literature studies, prior to any experimental work being done, liquid butane appeared to satisfy many of the above criteria.

Equipment selection

Since the process under consideration is meant to treat organic spills or leaks into groundwater and/or surface waters, and since these may be anywhere, the equipment must be transportable. Thus the process technology must lend itself to a configuration that can be carried on truck(s) or trailer(s). It must be rugged and capable of being set-up and started-up in a very short time under severe weather conditions - since spills cannot be scheduled for clement weather only.

The butane liquid-liquid extraction concept is believed to be ideally suited to meet these requirements. The solvent volume is relatively small, since it is recovered and recycled. The storage pressure is low even in hot weather. The system will not freeze in winter as can a steam-heated system. The entire process should be capable of a single truck or trailer mounting.

The key item of equipment - the contactor - is available in a wide variety of designs. The selection of this item must consider the extraction efficiency required and the dimension and weight requirements of each type. This aspect will be the subject of further study.

Universality

The process and solvent selected should be as universal as possible, so as to be capable of handling a range of organic contaminants. Since data are not available on K_D values for a wide range of solvents and organic contaminants, the selection of butane as the preferred solvent for cleaning-up water was a judgement decision based on limited information and limited previous experience (Won and Prausnitz, 1974). Other solvents may meet the criteria better than butane. However, butane does meet the criteria of:

- low toxicity (butane is actually approved by the US FDA as a general purpose food additive)

- low solubility in water

- low boiling point (-0.5° C)

- large density difference with water (should be easily separable by gravity settling).

Predictability

Ideally the process should be capable of mathematical modeling with the expectation that the results from the model should approximate actual results that would be achieved in the field. This requires knowledge of distribution coefficients (K_D) and solvent selectivity that can be obtained only through experimentation. These empirical data were expected to be obtained from the project, and were expected to provide the basis of a mathematical modeling of the butane stripping process.

4.1.4 Previous applications of solvent extraction.

Liquid-liquid extraction is reported to be used for the recovery of organic compounds in the following examples (Rousseau, 1987):

- recovery of acetic acid from dilute aqueous mixtures combining liquid-liquid extraction with azeotropic distillation

- separation of aromatic and aliphatic hydrocarbons in the petrochemical industry (the "Udex" system).

- recovery of penicillins from broth in the pharmaceutical industry.

Supercritical fluid extraction (SFE) is reported as being utilized to decaffeinate coffee beans, to recover hydrocarbon liquids from heavy oils, and to recover oxygenated hydrocarbons from water (Humphrey et al. 1984). Liquid carbon dioxide is used in large quantities in tertiary oil recovery from reservoirs.

Previous experimental work has been carried out by a number of researchers.

Won and Prausnitz (1974) examined the recovery from water of common organic solvents such as ketones, acetones, aldehydes and phenolics using 1-butane and i-butylene as solvents.

Barbari and King (1982) examined the extraction of chlorinated hydrocarbons and aromatics from water into undecane, and concluded that liquid-liquid extraction is potentially attractive for removing these contaminants from water.

Earhart et al (1976) reported on the extraction of pollutants from industrial wastewaters. The report concluded that although liquid-liquid extraction is used commonly to recover or purify organics from process streams, it is used only infrequently to treat wastewaters. However, it was also concluded that extraction of organics from aqueous streams using a volatile solvent was a promising and economic technique.

MacKay and Medir (1983) report on solvent extraction for industrial effluent treatment and describe 8 processes for the recovery of phenols from wastewaters. Numerous other liquid-liquid extraction processes are referred to, including the recovery of salicylic acid, fatty acids, oil and acetic acid from wastewaters.

Reynolds (1983) reported on the extraction of pesticides from process streams using high volatility solvents including hexane and pentane. Extraction efficiencies of 99.9999% were reported.

Witt and Forbes (1971) describe a number of applications for solvent extraction in industry. Applications discussed are: refinery foul water treatment; desalination; penicillin recovery; phenol recovery. Their conclusion was "...The future of solvent extraction in aqueous waste processing systems seems bright."

The evidence provided by the literature is sufficient to indicate that solvent extraction is worth careful consideration. Results obtained by some researchers show very high extraction efficiencies and/or high K_D values that augur well for the development of a successful processing system.

4.1.5 Extraction devices

So many types of liquid-liquid extraction devices have been developed or are in use that it is impossible to describe them all in a brief report. Only the main types of devices will be touched upon, especially those most applicable to the butane stripping process, and those that influenced the design of the test apparatus used in the tests discussed in this report.

Extraction processes may be batchwise or continuous systems. In batch systems, the extraction takes place in a single vessel between fixed charges of water and solvent, after which the phases are settled and separated, or they may be removed and separated in an external vessel; fresh charges may then be introduced into the contacting vessel, or the water may be contacted with fresh solvent. In continuous systems, the water is contacted continuously with a flowing stream of solvent, with the two streams usually flowing countercurrent or co-current to each other.

Batch processes include mechanically-agitated mixing vessels in which the charge of contaminated water is added to a charge of solvent. After a fixed mixing period the agitator is stopped and the phases are allowed to separate. One variation on this process is the boiling of soap in kettles with lye. The mixing is created by steam injection and steam coils, while the lye is circulated through the soap using pumps to extract the glycerine.

For a system to treat contaminated waters in the field, we feel that a true batch system is unsuitable. However, a mixer-settler system, in which a series of mixer-settlers give several stages of extraction in series, offers high efficiency, simplicity and reliability, and relatively modest cost for cleaning-up environmental emergencies.

Mixer-settler systems include a mixing chamber for intimate contact of the solvent and feed streams, followed by a settling chamber for phase separation. (each mixing chamber and settler forms a Stage). Mixer-settlers offer high stage efficiency, reliable scale-up and good operating flexibility. In the specific case of butane-water, where emulsion formation is not a problem, mixer settlers with intensive mixing can give extremely high stage efficiency, and for contaminants with high K_D values a small number of stages can give excellent decontamination of waters.

Continuous systems include: spray extractors; mechanically agitated extractors; packed columns; plate and tray columns; centrifugal units. These are discussed briefly below:

Spray extractors

Spray extractors are simply vessels in which the phases are introduced into the top (dense phase) and bottom (light phase) via spray nozzles. As the two phases pass each other, there is an exchange of constituents, with the solvent phase removing organic from the aqueous phase. Because the contact is solely due to the countercurrent streams of bubbles contacting each other and the bulk liquid, extraction efficiency is low.

Mechanically agitated extractors

Rotary agitated columns include components that rotate around the vertical axis of a column. Baffles are often inserted within the column to avoid vertical mixing. Typical proprietary columns are: 1) York-Scheibel column - providing wire mesh coalescers; 2) Rotating Disk Contactor - flat disks on a central shaft; 3) Oldshue-Rushton Column - uses turbine impellers and compartments separated by horizontal stator-ring baffles; 4) Kuhni contactor - contains a shrouded turbine impeller; 5) Asymmetric Rotating Disk - the rotating disks are mounted off-centre in the column; 6) Graesser Contactor - a horizontal design using buckets to disperse the phases. Sketches of some of the above columns are contained in Figure 1.

Reciprocating-plate columns include components that reciprocate or vibrate along the vessel axis. A typical proprietary column is the Karr Contactor, which contains a series of perforated disks set on a central shaft. The shaft is reciprocated, imparting a "pumping" action to the disks. Fluid flows through the disk perforations and is mixed by the jetting action as it leaves the disk. A sketch of a Karr contactor is shown in Figure 2.

Packed columns

The packed column extractor is a vertical column containing a standard packing (e.g. saddles, rings, or structured packings of the gauze or mesh type). They are efficient contactors of the liquid phases, although they are subject to flow restrictions because they will flood at some given flowrate. A diagram of a packed column is given in Figure 3.

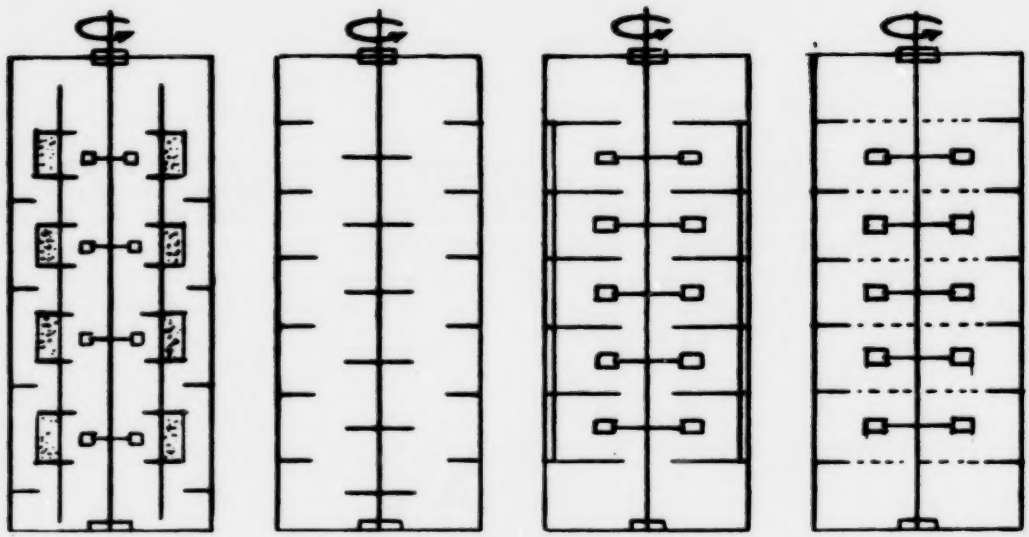


Figure 1 - Rotary agitated columns

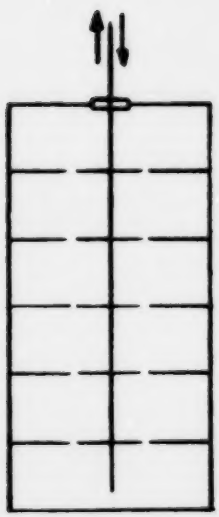


Figure 2
Reciprocating-plate column

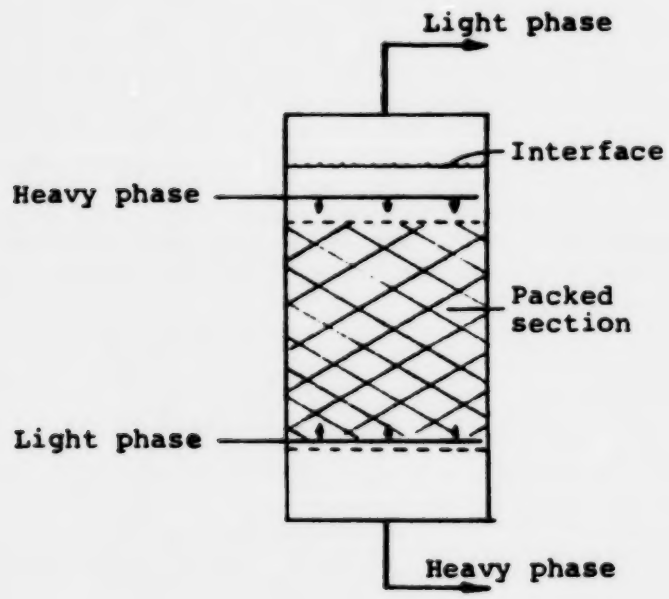


Figure 3
Packed column

Plate and tray columns

The sieve tray column is similar to those used in distillation. Large downcomers are needed to allow the denser layer to descend, while tray perforations are larger than normal to allow the lighter phase to rise. There is little published information on the performance of these units for liquid-liquid extraction. A variation on this column is the Controlled-Cycling-Tray Extractor, in which feed and solvent streams are alternately turned on/off, and the flow through the tray perforations causes intense mixing.

Centrifugal contactors

The Podbielniak contactor is typical of these devices. They operate somewhat like centrifuges, with the difference that fluid density differences cause the fluids to flow in opposite directions by the centrifugal force. They give rapid contacting, and are very compact. They are often used in the pharmaceutical industry, where unstable materials are being processed.

4.2 Program design

The literature review provided the information on which to design an experimental program to yield results we could use to establish the worth of butane stripping as an environmental clean-up technique. An integral aspect of the program was the design and construction of a small-scale contactor where the extraction would take place.

One other important benefit from the literature review was the information obtained on distribution coefficients (K_D) from previous experimental work. These data meant that we were not completely in the dark regarding the likely order of magnitude for some of the compounds to be tested.

The data were combined to create a program of experimental work that would have unassailable methodology and would provide definitive results. The program of experimental work is described in Section 5 following.

5. EXPERIMENTAL WORK

5.1 Introduction

The test program was devised to provide the maximum amount of data at the lowest cost. It was also aimed at utilizing a test apparatus that could be related to a field installation via appropriate scale-up factors. Since Phase II of the project (if it continues) is to undertake more test work in a "mini" pilot-plant operating continuously, the test program was also aimed at providing relevant data for developing Phase II.

5.2 Description of the test apparatus

An agitated contactor vessel was made from a 80-95 mm od (tapered) x 150 mm long x 10 mm thick Corning glass tube, with a design pressure of 50 psig (446 kPa). The ends of the tube were covered with 5 mm thick stainless steel end plates sealed by teflon gaskets and tensioning bolts. The contactor vessel is shown in photographs 1 and 2. A pressure gauge and safety relief valve were fitted to the vessel.

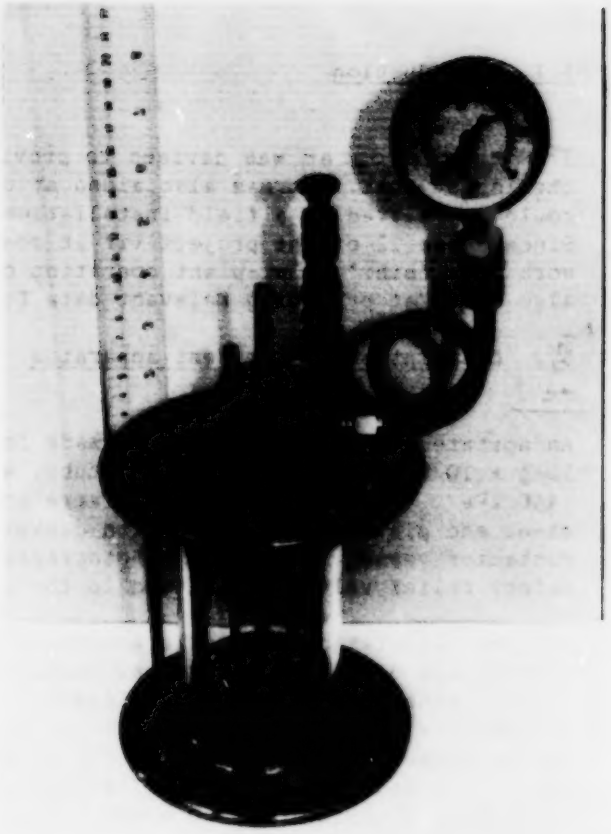
An internal stationary baffle arrangement was inserted into the contactor (photograph 3) to prevent vortex formation and to enhance mixing. A turbine mixer with two mixing elements (photograph 3) was inserted into the contactor via a sealed opening in the top stainless steel end plate. The mixing elements were adjustable for height, and were designed to rotate in the butane and water layers. The mixer was driven via a flexible drive cable from a variable speed motor. Mixer speed was variable from about 10 rpm to 320 rpm.

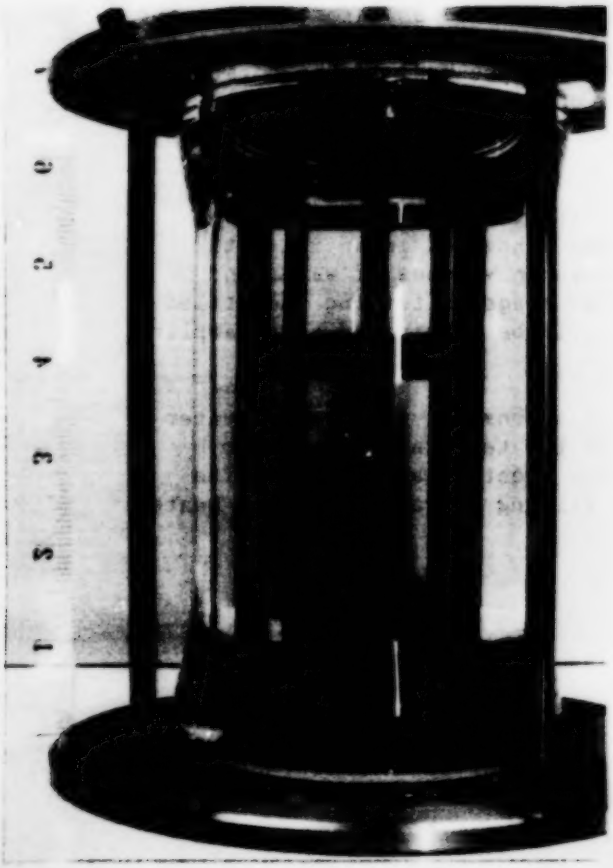
Water and butane were introduced into the contactor via a 1/4" swagelok fitting in the top end plate. The butane was condensed in an ice-bath before it was added to the contactor. When known volumes of water and butane had been added to the vessel, the 1/4" swagelok fitting was sealed by a high pressure septum assembly. This could be penetrated by a sampling needle without leakage.

The entire contactor vessel was submerged in a constant temperature water bath (photograph 4) to give close control of the temperature and also, secondarily, to act as a safety shield in the event of a breakage. The contactor was kept at about 0°C during the filling operation and was heated to 20°C for each test.

Photograph 1.

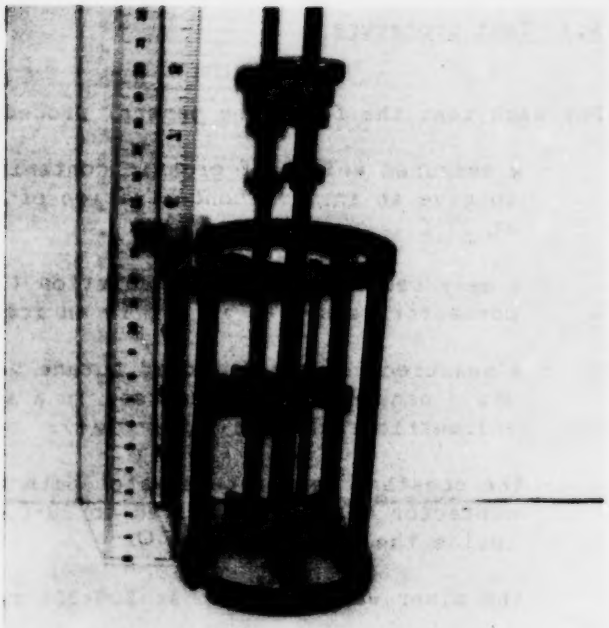
Overview of test apparatus showing sampling septum and pressure relief assembly.





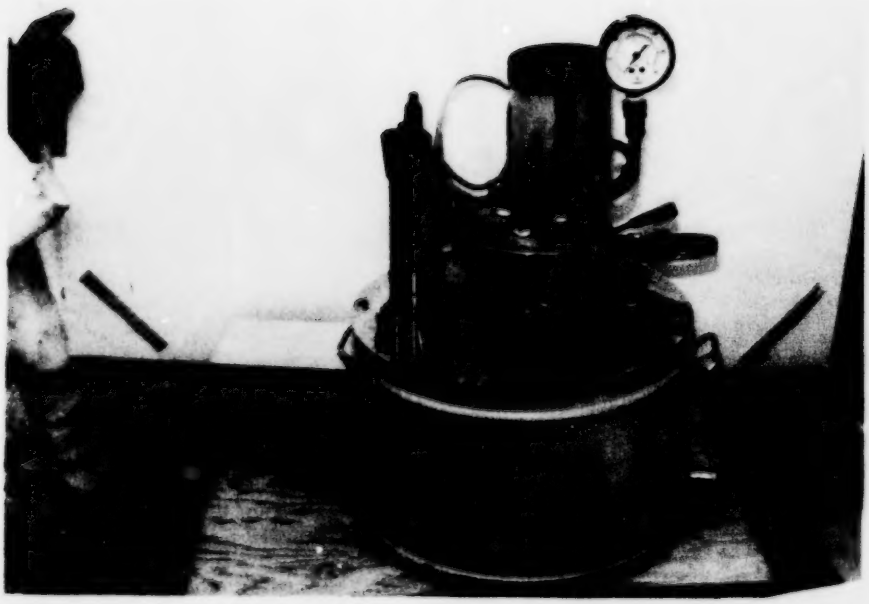
Photograph 2.

Close-up of apparatus showing construction details



Photograph 3.

Close-up of internal baffle arrangement and agitator assembly.



Photograph 4.

Complete assembly in water bath.

5.3 Test procedure

For each test the following general procedure was adopted.

- a measured weight of organic contaminant was added to distilled water to give an initial concentration of about 50% of its solubility limit at 25°C.
- a measured volume of this solution (250 ml) was added to the contactor vessel as it sat in an ice bath.
- a measured volume of liquid butane was transferred to the contactor via a condenser coil immersed in a salt/ice mixture (the butane/water volumetric ratio was 1/5 or 1/1).
- the constant temperature water bath heater was turned on and the contactor vessel was heated to 20°C, as measured by a thermocouple inside the vessel.
- the mixer was turned on at 300-320 rpm and the stopwatch was started.
- at fixed intervals the mixer was stopped, the butane and water layers were allowed to separate (@ 2 mins), and water and butane samples were withdrawn using a hypodermic syringe with a long needle (15 cm). The butane sample was injected into a sample vial containing a known weight of heptane as absorbent.
- samples of the water layer and of the heptane were analyzed within 10 minutes after sampling by gas chromatography (GC). Each sample was analyzed 4-8 times, with the individual results being combined to give a mean value.

5.4 Test difficulties.

A number of technical difficulties were encountered during the tests. Most of them related to the problems of handling liquid butane in the small-scale batch apparatus utilized, and would not necessarily be a concern in a continuous or larger-scale system. The technical problems encountered were:

Butane liquefaction

Butane was obtained in a 20 kg bottle delivering gaseous butane via a 2-stage gas pressure regulator. It was not possible to draw liquid butane directly from the bottle. This required running the gaseous butane through a stainless steel coil immersed in a salt/ice mixture to condense out liquid butane. The condensed butane was then transferred as liquid directly to the test apparatus which was maintained at 0°C during the filling stage.

Although seemingly a minor point, the need to liquefy the gaseous butane added about an hour of extra work to every test.

Butane solubility

At the test conditions (20°C and 120 kPa) butane dissolves in the water layer to the extent of about 1,000 ppm. The dissolved gas is released from solution when the pressure is released. During the analyses, butane gas bubbles tended to form on the GC micro-syringe, causing difficulties for the analyst in obtaining the precise injection volume needed. This slight problem was another source of extra work that affected every test, requiring many repetitions of each analysis.

Butane evaporation

At the test conditions in the contactor, butane is a liquid. As soon as a sample is withdrawn and pressure is released the butane volatilizes and cannot be analyzed. Therefore it is necessary to maintain the butane at as low a temperature as possible during the sampling stage. This difficulty was overcome by drawing the butane sample into an insulated syringe that had been pre-cooled in liquid nitrogen.

After drawing the sample into the cold hypodermic syringe, it was immediately injected into a known weight of heptane which retained the extracted organic contaminant.

The procedure adopted made it possible to sample the butane layer without too much difficulty. However, this last modification also had the effect of increasing the complexity and time required for each test.

In summary, the above problems, although solved and having little effect upon the result of the test program, served to increase the time requirement for each test from an estimated 2 hours to about 6 hours, and allowed only one test to be completed per day, instead of the several tests scheduled originally.

5.5 Test program

Environment Canada supplied a list of priority pollutants they wished to be tested with the butane extraction process. Based on the information supplied, the following test program was conducted:

Pollutant	Concentration (Note 1)	S/W (Note 2)	Analysis (Note 3)
Ethylene dichloride	4,225 ppm	1/1	GC
Ethylene dichloride	4,225 ppm	1/5	GC
Trichloroethane	639 ppm	1/5	GC
Tetrachloroethane	88 ppm	1/5	GC
Dichlorobenzene	74 ppm	1/5	GC
Benzene	960 ppm	1/5	GC
Phenol	4.0% w/v	1/1	GC
Phenol	4.0% w/v	1/5	GC
Methanol	10.1% w/v	1/5	GC
Methyl ethyl ketone	10.0% w/v	1/5	GC

Note 1: Concentration used is approximately half the solubility limit in water, except for methanol which has infinite solubility. Concentration in ppm refers to parts per million by weight.

Note 2: Butane/water ratio on volume basis.

Note 3: GC - gas chromatography

The laboratory test program commenced on 18 November, 1987, with a preliminary extraction of ethylene dichloride using butane/water in the ratio 1/1 (250 ml butane and 250 ml water + contaminant). After the first three runs the mixer shaft seized in its bushing and needed replacement. Subsequently, all further tests were completed without delays caused by equipment malfunction.

The initial tests, using ethylene dichloride as a model pollutant, showed excellent extraction into the butane phase, with K_D values of over 40. The initial ethylene dichloride concentration in the water of 4,300 ppm was reduced to about 100 ppm in a single 5 minute extraction. This was not an unexpected result, since it was predicted that non-polar organics would show a great affinity for the liquid butane solvent.

Subsequent tests with other non-polar organics confirmed the high degree of extraction that could be obtained with butane as the solvent. All test results are detailed and discussed in Section 6. of this report.

6. TEST RESULTS

6.1 Introduction

Details of the analytical techniques used and results obtained are discussed in this section of the report. Detailed analysis sheets are available on request, but are not included with this report.

6.2 Summary

High extraction efficiencies were obtained for the non-polar organic compounds tested. In all cases the K_D values calculated from our test results were of the same order of magnitude as those reported in the literature or as predicted by us prior to the test program. Measured and predicted K_D values are shown in Table 1.

Table 1

Measured and predicted values for K_D

Compound	Predicted (Note)	Measured
Ethylene dichloride *	None	43-47
Trichloroethane *	>414	592
Tetrachloroethane *	None	251
Perchloroethylene *	>2,700	3,222
Dichlorobenzene *	>3,030	2,918
Benzene *	400	216
Methanol	None	1.5
Methyl ethyl ketone	1.35-1.4	1.3-1.5
Phenol	0.2	0.15-0.23

Note: Predicted values were those either measured previously by other researchers (MEK; phenol), estimated from data in the literature (benzene), or "guesstimated" from data on extraction into undecane in the literature (dichlorobenzene; trichloroethane; perchloroethylene. Where no data were available to make a prediction possible this is shown as "none" in Table 1.

The compounds marked with an asterisk (*) have such high K_D values that stripping into butane in 3-4 stages should give 99.9999% extraction efficiency with ease. Butane stripping should be considered as the preferred method for removing these and similar chemicals from water.

Extraction efficiency from a single extraction stage was very high for non-polar organics, as shown in Table 2.

Table 2

Water layer concentrations and removal efficiency from a single stage extraction with n-butane.

Compound	$C_{Initial}$	C_{Final}	% Removal
Ethylene dichloride *	4,225 ppm	87	97.9 (2)
Ethylene dichloride *	4,225 ppm	728	82.8 (1)
Trichloroethane *	639 ppm	6.96	98.9 (1)
Tetrachloroethane *	88 ppm	1.80	98.0 (1)
Perchloroethylene *	93.4 ppm	0.17	99.8 (1)
Dichlorobenzene *	74 ppm	0.13	99.8 (1)
Benzene *	960 ppm	24.2	97.5 (1)
Methanol	10.1% w/v	7.82	22.6 (1)
Methyl ethyl ketone	10% w/v	7.54	24.6 (1)
Phenol	4% w/v	3.77	5.8 (1)
Phenol	4% w/v	3.49	12.8 (2)

Note: (1) Butane/water ratio 1/5; (2) Butane/water ratio 1/1.

The potential for removal of the organic compounds marked with an asterisk (*) is clear from the above tabulations. Comparable extraction efficiency should be obtainable from each stage of a multi-stage contactor.

6.3 Experimental and analytical procedures

Experimental procedure

The model compound was weighed out using an analytical balance and transferred quantitatively to a 1,000 ml volumetric flask containing about 800 ml distilled water. The solution was homogenized in an ultrasonic bath at 20-23°C for 30 minutes to complete dissolution. The solution was then made up to 1,000 ml. In general, concentrations corresponding to about 40-60% of the literature solubility data were used.

500 ml of the freshly prepared solution were transferred to the extraction vessel (contactor) and cooled to 0-4°C in a constant temperature bath without stirring the solution. Liquid n-butane was transferred to the contactor from the main storage tank via a stainless steel condensing coil maintained at about -5°C in an ice/salt bath. After metering the required volume of liquid n-butane into the contactor, the contactor and two-phase liquid were heated to 20°C in the constant temperature bath without stirring (about 15 minutes).

Liquid levels of the two phases were recorded prior to starting the extraction. The speed of the dual turbine mixer blade was maintained at about 320 rpm throughout each test, with speed being checked by a strobe tachometer. At set intervals (2, 5, 10, 15, 30 minutes) the agitation was stopped and the phases were allowed to separate.

Aqueous phase samples (6-10 ml) were taken via the septum port using a long hypodermic needle (length 15 cm). These samples presented no problems. Sampling the butane layer was difficult, because it volatilizes rapidly once pressure is released. Ultimately we developed a procedure for the butane sampling using a syringe pre-cooled in liquid nitrogen. About 3-4 ml of butane were withdrawn and transferred into a vial containing a weighed amount of n-heptane (≈10 ml) as the absorbent. Therefore, the weight of the butane layer collected could be weighed accurately. Liquid butane sampling was confined to the end of each test as a check on the aqueous phase analyses.

Although each extraction test appears to take only 15-30 minutes, the actual elapsed time for each test was about 3 hours, since it also covered the preparation time, and the sampling was time-consuming.

Analytical procedure

Samples were analyzed using a Varian Vista gas chromatograph (GC) equipped with a flame ionization detector (FID). Operating conditions in the GC were:

- carrier gas: UHP grade argon gas; flow rate 30 ml/min
- capillary silica column DB-5 (id 200 µm x 25 m)
- detector sensitivity range: 10^{-12}
- column temperature: isothermal, depending on the model compound, range 33 to 80°C.

2 or 8 µl per injection was used for each analysis. Up to 8-12 analyses per sample were conducted in order to obtain a set of 4-8 good results for averaging. Calibration was performed prior to the sampling analysis using the original fresh solution. In some cases, calibration was also cross-checked with standard solutions of the model compound in n-heptane.

6.4 Detailed test data

6.4.1 Introduction

The analytical results presented in the following sections are the mean values derived from replicate analyses (4-8) performed on each sample. These mean values are then summed to give average values, which are those presented in Tables 1 and 2 above. It will be noted that individual values for each time interval may not agree exactly with the values in Tables 1 and 2; this is the result of averaging.

6.4.2 Ethylene dichloride - Tests 1 to 4

Ethylene dichloride (EDC) was the first model compound tested. As such, several tests were made while we checked out and debugged the test apparatus. The tests included:

Test 1. Initial EDC concentration 835 mg/l at a nominal butane/water (B/W) volumetric ratio of 1/1. Temperature 20°C. Extraction was rapid, reaching equilibrium within 2 minutes. EDC concentrations in the water layer were measured by GC. EDC concentrations in the butane layer were calculated from the mass balance. Results are presented below.

Time (mins)	EDC in water	EDC in butane	K_D *	Removal
0	835 ppm	--	--	--
2	19.1	816	42.7	97.7%
5	17.0	818	48.1	98.0%
10	20.9	814	40.0	97.1%
25	19.2	816	42.5	97.7%
			Mean 42.5	

* All K_D values are calculated from measured concentrations.

Test 2. Initial EDC concentration 4,225 mg/l at a nominal B/W ratio of 1/1. Temperature 20°C. Extremely rapid extraction, as in Test 1.

Time (mins)	EDC in water	EDC in butane	K_D	Removal
0	4,225 ppm	--	--	--
2	92	4,133	44.9	97.8%
5	86.9	4,138	47.6	97.9%
Mean	89.5	4,136	46.3	97.9%

Test 2 was terminated when the mixer shaft seized in the top bushing. This test was witnessed by Ms. F. Laperriere of Environment Canada. Repairs to the mixer and modifications to the apparatus took about a week to complete.

Test 3. Initial EDC concentration 4,225 mg/l at a nominal B/W ratio of 1/5. Temperature 0°C. This test was undertaken to evaluate the effectiveness of liquid butane extraction at low temperature. Extraction efficiency dropped, although good extraction would still be possible in a multi-stage apparatus.

Time (mins)	EDC in water	Butane in water *	K_D	Removal
0	4,225 ppm	--	--	--
2	676	934	26.3	84.0%
5	695	885	25.4	83.6%
10	728	795	24.0	82.8%
		Mean	25.2	

* Butane in water concentrations are ±30% because of the difficulty with gas bubble formation on the GC injection syringe.

Test 4. Initial EDC concentration 4,402 mg/l at a nominal B/W ratio of 1/5. Temperature 20°C

Time (mins)	EDC in water	Butane in water	K_D	B/W * ratio	Removal
0	4,402 ppm	--	--	--	--
2	893	863	20.5	5.2	79.7%
5	592	999	41.8	6.5	86.6%
10	779	1,187	47.8	10.3	82.3%
15	907	1,151	46.7	12.1	79.4%

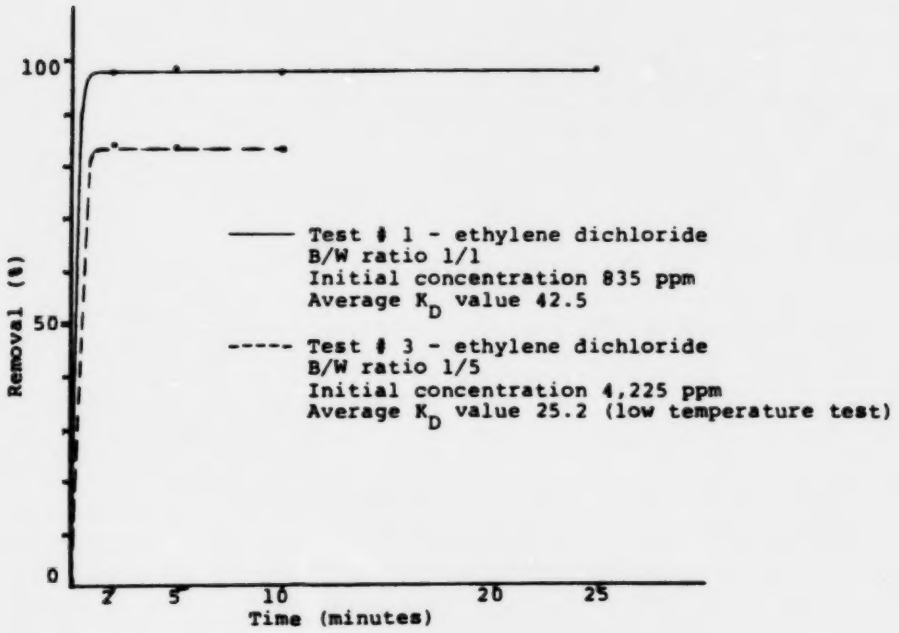
Removal efficiency Vs extraction time for Tests 1 and 3 is shown on Figure 4.

* See Note on following page above Figure 4

Figure 4.

Ethylene dichloride removal from water with time. Test 1 was with a butane/water ratio of 1/1. Test 2 was with a butane/water ratio of 1/5. Note the extremely rapid removal, which is typical of many of the tests. Samples were not taken prior to 2 minutes, so the shape of the curve to that time is estimated.

Note: It was noticed during Test 4 that the withdrawal of a butane sample and a water sample affected the relative ratio of butane and water (the B/W ratio). The change in B/W ratio is shown for all subsequent Tests. The nominal B/W ratio is the approximate volumetric ratio at the start of each Test.



6.4.3 1,1,1-trichloroethane - Test 5

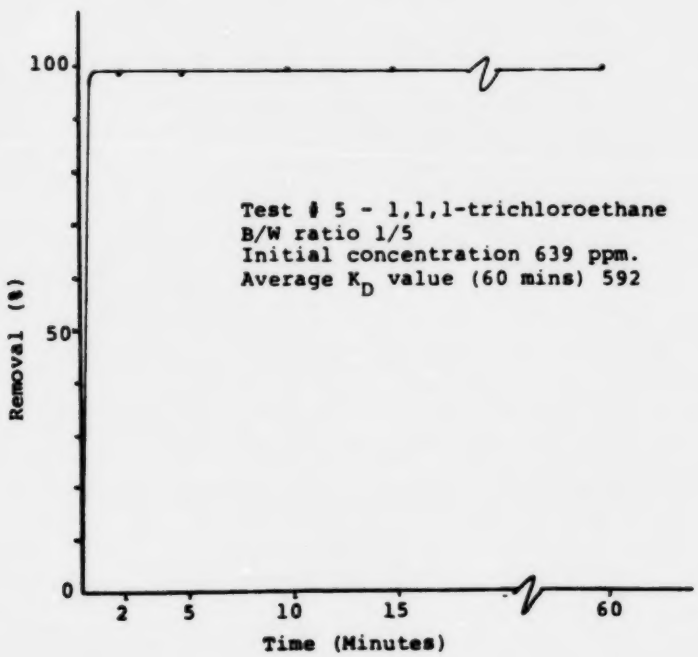
Initial TCE concentration 639 mg/l at a nominal B/W ratio of 1/5. Temperature 20°C.

Time (mins)	TCE in water	Butane in water	K_D	B/W * ratio	Removal
0	639 ppm	--	--	--	--
2	11.2	347	288	5.13	98.2%
5	10.8	628	341	5.8	98.3%
10	6.35	505	598	6.0	99.0%
15	7.62	677	516	6.22	98.8%
60	6.90	680	592	6.47	98.9%

* As noted previously, the B/W ratio changes during each test because the volume of water sample removed is a measureable quantity of the total liquid in the contactor. Also, butane dissolving in the water gives an apparent change in the ratio.

Figure 5

Trichloroethane removal with time. Extremely rapid removal and a high K_D value.



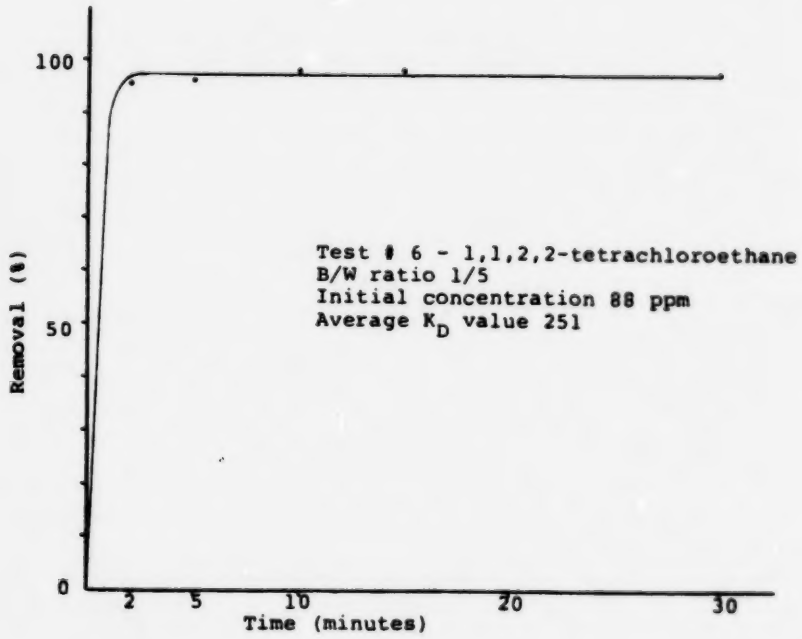
6.4.4 1,1,2,2-tetrachloroethane - Test 6

Tetrachloroethane (TetCE) was tested because a mistake was made in delivery by the supplier, and we received TetCE instead of 1,1,2,2 tetrachloroethylene (perchlorethylene). Since the material was on hand, and since it is a Priority Pollutant, we decided to include an additional unscheduled test to our program and to test TetCE. Initial TetCE concentration 88 mg/l at a nominal B/W ratio of 1/5. Temperature 20°C.

Time (mins)	TetCE in water	Butane in water	K_D	B/W ratio	Removal
0	88 ppm	--	--	--	--
2	3.76	--	112	5.0	95.7%
5	3.33	--	132	5.2	96.2%
10	1.66	--	265	5.09	98.1%
15	1.68	580	267	5.19	98.1%
30	2.07	844	221	5.35	97.6%

Figure 6

Tetrachloroethane removal with time.



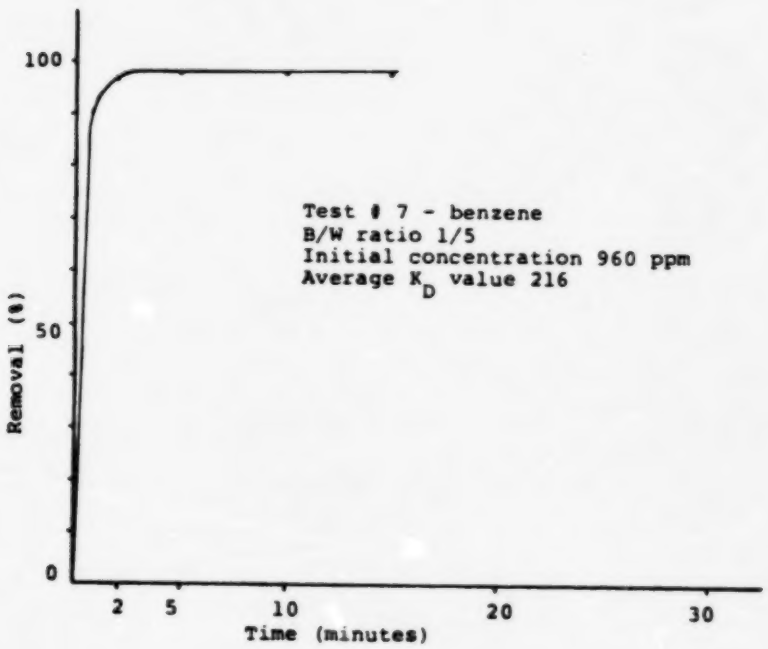
6.4.5 Benzene - Test 7

Initial benzene concentration 960 mg/l at a nominal B/W ratio of 1/5. Temperature 20°C.

Time (mins)	Benzene in water	Butane in water	K_D	B/W ratio	Removal
0	960 ppm	--	--	--	--
2	34.2	1,529	151	5.57	96.4%
5	23.3	1,476	220	5.47	97.6%
10	24.5	--	218	5.70	97.4%
15	24.8	--	209	5.55	97.4%

Figure 7

Benzene removal with time. Curve shape below 2 minutes estimated.



6.4.6 1,2-dichlorobenzene - Test 8

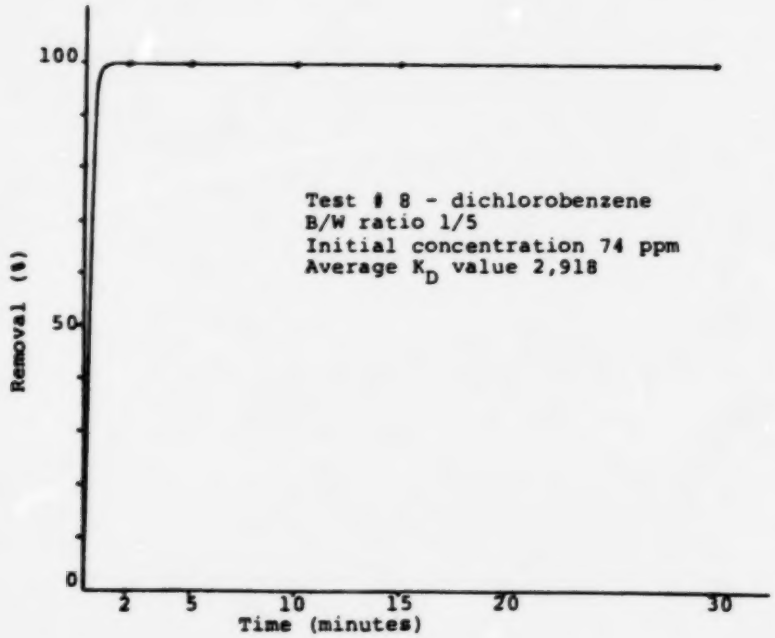
Initial DCB concentration 74 mg/l at a nominal B/W ratio of 1/5.
Temperature 20°C.

Time (mins)	DCB in water	Butane in water	K_D	B/W ratio	Removal
0	74 ppm	--	--	--	--
2	0.35	716	939	4.46	99.5%
5	0.19	--	1,803	4.6	99.7%
10	0.12	--	2,783	4.52	99.8%
15	0.15	--	2,298	4.67	99.8%
60	0.117	--	2,894	4.58	98.9%

Because of the very low concentrations of DCB being measured and the injection volume (8 µl), the GC was operating close to the detection limit, with a large potential error. A sample of the butane layer was taken at the end of the test and analyzed for DCB. From 3 replicate analyses on the butane layer, the K_D values were calculated as 2,783, 2,894 and 3,078, giving an average of 2,918.

Figure 8

Dichlorobenzene removal with time.



6.4.7 Methanol - Test 9

Initial methanol concentration 10.1% w/v at a nominal B/W ratio of 1/5. Temperature 20°C.

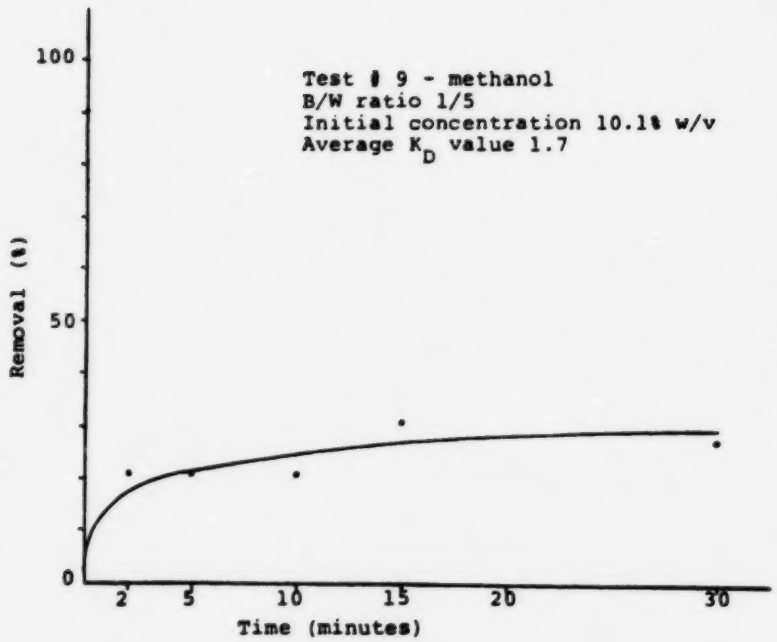
Time (mins)	MeOH in water	Butane in water	K_D	B/W ratio	Removal
0	10.1 %	--	--	--	--
2	7.95	(1)	1.38	5.11	21.3%
5	7.99	(1)	1.36	5.16	20.9%
10	8.03	(1)	1.41	5.45	20.5%
15	6.98	(1)	2.38	5.33	30.9%
30	7.32	(1)	1.98	5.24	27.5%

A mean K_D value of 1.5 was derived by discarding the 15 minute test result, since it appears to be too high.

Note 1. Butane in water was not obtained because the butane peak overlapped the MeOH peak on the GC.

Figure 9.

Methanol removal with time.



6.4.8 Methyl ethyl ketone - Test 10

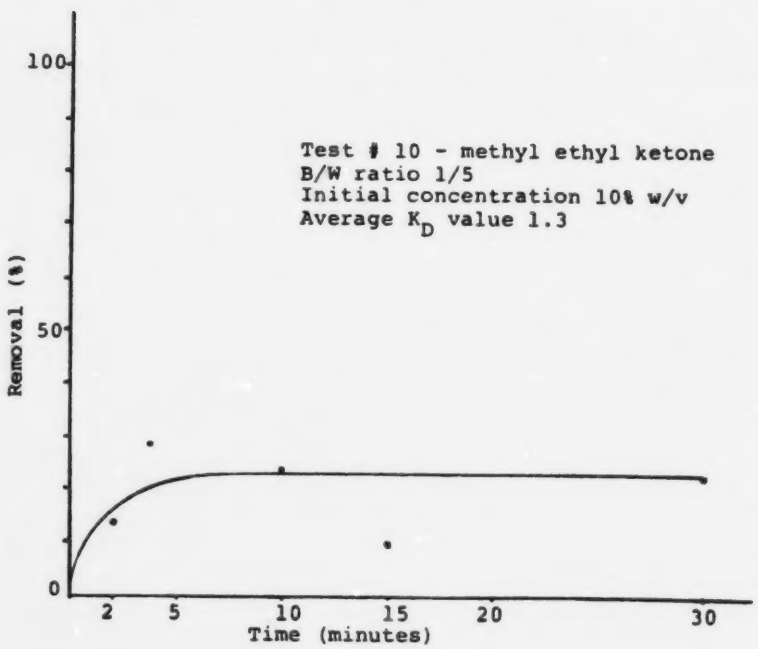
Initial MEK concentration 10% w/v at a nominal B/W ratio of 1/5.
Temperature 20°C.

Time (mins)	MEK in water	Butane in water	K_D	B/W ratio	Removal
0	10.0 %	--	--	--	--
2	8.65	(1)	0.75	4.79	13.5%
5	7.18	(1)	1.55	3.96	28.2%
10	7.63	(1)	1.19	3.82	23.7%
15	9.06	(1)	0.41	3.89	9.4%
30	7.81	(1)	1.09	3.81	21.9%

Note 1: Butane in water could not be measured because of interference with the MEK in the GC.

Figure 10

Methyl ethyl ketone removal with time. Considerable scatter of the data points makes the curve-fitting uncertain. The 15 minute result has been neglected for the sake of fitting a reasonable curve.



6.4.9 Phenol - Tests 11 and 12

Test 11. Initial phenol concentration 4.0% w/v at a nominal B/W ratio of 1/5. Temperature 20°C.

Time (mins)	Phenol in water	Butane in water	K_D	B/W * ratio	Removal
0	4.0 %	--	--	--	--
2	3.91	1323 ppm	0.11	4.92	2.3%
5	3.73	805	0.38	4.85	6.8%
10	3.69	987	0.41	4.89	7.8%
15	3.64	938	0.48	4.87	9.0%
30	3.90	1133	0.13	4.89	2.5%

These analyses were double checked. One sample of butane was absorbed in n-heptane which was then analysed for phenol. From this analysis, K_D was calculated at 0.17. A gravimetric analysis was run on a second sample, from which K_D was calculated at 0.12. From these data, we feel that the value of 0.20 reported in the literature (Earhart et al, 1977) can be treated as essentially correct.

Test 12. Same as Test 11, except the B/W ratio was increased to 1/1.

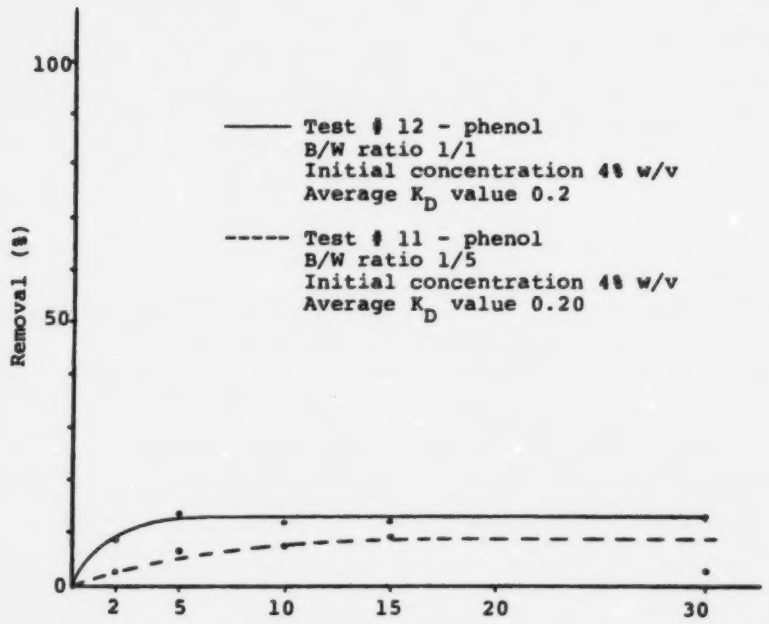
Time (mins)	Phenol in water	Butane in water	K_D	Removal
0	4.00 %	--	--	--
2	3.67	826 ppm	0.09	8.3%
5	3.45	570	0.16	13.8%
10	3.53	1265	0.15	11.8%
15	3.52	1062	0.14	12.0%
30	3.49	1410	0.15	12.8%

Analyses were checked as in Test 11. From analysis of phenol in Heptane, K_D = 0.23. From gravimetric analysis, K_D = 0.16.

Figure 11.

Phenol removal with time. Test 11 was with a butane/water ratio of 1/5. Test 12 was with a butane/water ratio of 1/1. Low removal efficiency indicates that butane stripping is not suitable for removing phenol from water.

(High removal efficiency may be possible with a dual solvent process, although this was not examined in these tests. Theoretical extraction of 99.99% in 6 stages is possible using dual solvents).



6.4.10 Perchloroethylene - Test 13

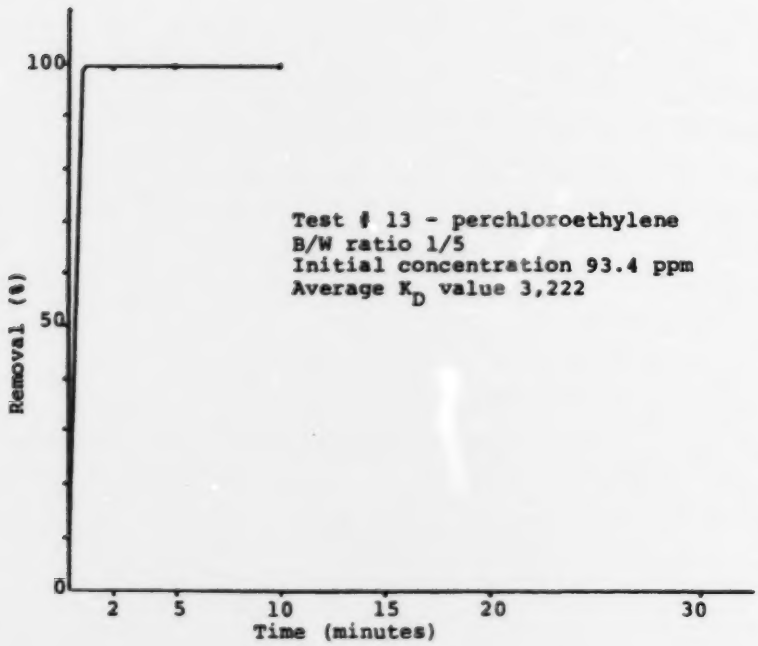
Initial PCE concentration 93.4 mg/l at a nominal B/W ratio of 1/5. Temperature 20°C.

Time (mins)	PCE in water	Butane in water	K_D	B/W * ratio	Removal
0	93.4 ppm	--	--	--	--
2	0.649	1006	699	4.89	99.3%
5	0.479	1038	874	4.91	99.5%
10	0.545	1151	3,222	5.56	99.4%

Because the GC was operating close to the detection limit. large variations and errors were possible, requiring multiple analyses. A check analysis was run on the butane layer, from which it was determined that the PCE concentration in the butane was 1,756 mg/l, giving a calculated K_D value of 3,222 (1,756/0.545). From this result, the calculated concentration of PCE in the water layer at equilibrium is 0.166 mg/l.

Figure 12

Perchloroethylene removal with time. Extremely rapid removal, with almost 100% removed within 2 minutes. Highest K_D value obtained in the tests.



6.5 Butane solubility in water

It is apparent from the preceding data that butane has considerable solubility in water when mixed in the liquid phase at elevated pressure. It also appears that the nature of the contaminant being extracted affects the butane solubility. Butane solubility ranged from about 1,500 mg/l (Test 7) to about 600 mg/l (Test 5). We assume that the organic contaminant has some effect on the butane solubility, although we were unable to conduct investigations into the phenomenon. (liquid butane was equilibrated with distilled water in the absence of any other organic, giving a mean concentration of about 4,000 mg/l)

Regardless of whether the concentration is 600 or 1,500 mg/l, these are excessive concentrations in terms of economic losses to the process, and in terms of the environmental injury to aquatic organisms. It is necessary to reduce the butane concentration to an acceptable level before water can be discharged to any receiving water.

Preliminary tests undertaken with a simple laboratory set-up indicate that butane concentration can be reduced from over 1,000 mg/l to below 50 mg/l by trickling the water over expanded polystyrene packing. In our tests, a small quantity of expanded polystyrene chips in a small container were shaken with water saturated with butane. After about 5 minutes at room temperature (20°C) the butane concentration had fallen from over 4,000 mg/l to about 70 mg/l. Further sparging with oxygen for 2 minutes reduced the concentration to less than 50 mg/l.

In another test, shaking a butane-saturated water sample with a fine silica powder (40 micron) displaced butane from solution rapidly, leaving a residual concentration of 20-30 mg/l after 5 minutes. Clearly, therefore, dissolved butane can be removed from solution if a suitable surface is provided. This subject requires further investigation to optimize the butane removal conditions.

In a properly designed stripping column, operating at reduced pressure and with continuous removal of gaseous butane, we would expect to consistently reduce the dissolved butane concentration to less than 50 mg/l, and possibly even lower. This stripped water would then pass through an air stripper to further reduce the butane to about 5 mg/l before the water was returned to its source or to another receiver.

The limited tests we have conducted are sufficient to convince us that butane recovery is feasible, and that butane solubility in the water is not a limitation to the process. The traces of butane remaining in the water should have no impact on non-aquatic uses, such as irrigation water. Bio-assays will show if low concentrations of butane cause any toxicity or morbidity in rainbow trout fingerlings or other test organisms. Taste tests will show if butane has a discernible effect on potable water supplies.

6.6 Butane recovery from organic pollutants

Once the organic pollutant has been extracted from the water it is necessary to separate the butane from the pollutant with a high degree of efficiency, so as to recycle very pure butane back to the stripping unit. The pollutant must be reduced to a very low concentration in the butane if it is to remove 99.99+% of pollutants from water.

We consider that fractionation in a multi-stage column is the preferred method to separate the butane from the organic pollutant. Butane has a much lower boiling point (-0.5°C) and higher volatility than almost all pollutants of concern. Separation by distillation should, therefore, be simple as long as azeotropes are not formed. We are not aware that butane forms azeotropes with other organics, and do not consider this to be a significant concern.

Westcoast Transmission Company Limited (WTCL) offered to run a number of trials on their process simulation computer ("HYSIM" by Hyprotech, Calgary). This allows the simulation and/or design of complex gas processing and refinery operations with a high degree of accuracy. The trials they undertook were to design a fractionation column to separate butane from a pollutant, with the following parameters:

Feed rate	100 kg/h
Feed composition (23.5°C/240 kPa)	99.5% butane 0.5% organic
Overhead product (5°C/125 kPa)	99.999% butane 0.001% organic
Bottom product (5°C/125 kPa)	80.0% butane 20.0% organic

Methanol and benzene were taken as example organic compounds, since they have similar vapour pressure/temperature characteristics to many of the other chemicals tested, and can be considered as representative.

The computer simulations indicated that the methanol separation can be achieved with 9 trays, and the benzene separation can be achieved with 8 trays, in a tray-type fractionation column with reflux. If this is related to a packed column, with a high-efficiency packing, similar results can be obtained from a relatively compact column less than 3 m in height. This is easily transportable, and could be shipped lying flat if required. Specific design details are the subject of future work.

The calculated net heat input to the fractionation column agrees closely with that assumed by us in the energy balance for the process. This heat is being supplied to maintain the distillate at 5°C, and is available from

many heat sources, including the butane compressor and the engine-generator exhaust or cooling system. The reboiler duty varies with the organic pollutant, being 57,000 kJ/h for the methanol fractionation and 38,000 kJ/h for the benzene fractionation.

Copies of the computer outputs for the trials conducted by WTCL are contained in Appendix 1.

6.7 Performance prediction

Having a process that works is a significant benefit, but its operation must be predictable and consistent. With data on activity coefficients or on K_D values the extraction efficiency of a stage can be calculated. Unfortunately, little data exists on K_D values for the wide range of organic pollutants of environmental concern.

We have discovered a relationship that may hold true for a large number of organic compounds, by which an approximation of K_D value may be made for previously untested chemicals. The relationship is illustrated in Figure 13. A plot of log K_D against log S, where S is the solubility of the organic compound in water, gives a straight line with the equation:

$$\log K_D = 1.681 - 0.9934 \log S$$

Although phenol and methanol do not fit this line well, all the other organics tested fit remarkably well. If such a relationship can be demonstrated to have universal applicability, it will allow a prediction of K_D values and hence of extraction efficiency for any chemical. Additional laboratory work is necessary to increase the data base and to prove if there is a good correlation.

With data on K_D values it is possible to derive mathematical relationships between extraction efficiency and the number of theoretical extraction stages. This is illustrated on Figure 14, which shows the number of theoretical mixer-settler stages required to give 99.99% and 99.9999% extraction for varying K_D values. Clearly, high extraction efficiency can be achieved in a small number of stages for any organic having a K_D value greater than 5-10.

Further development of the theoretical basis for the extraction process and for predicting its operation requires additional laboratory work, which would be more research oriented than the proposed pilot-plant operation, i.e. that would seek K_D data and solubility data with the objective of establishing a butane stripping correlation for all Priority Pollutants.

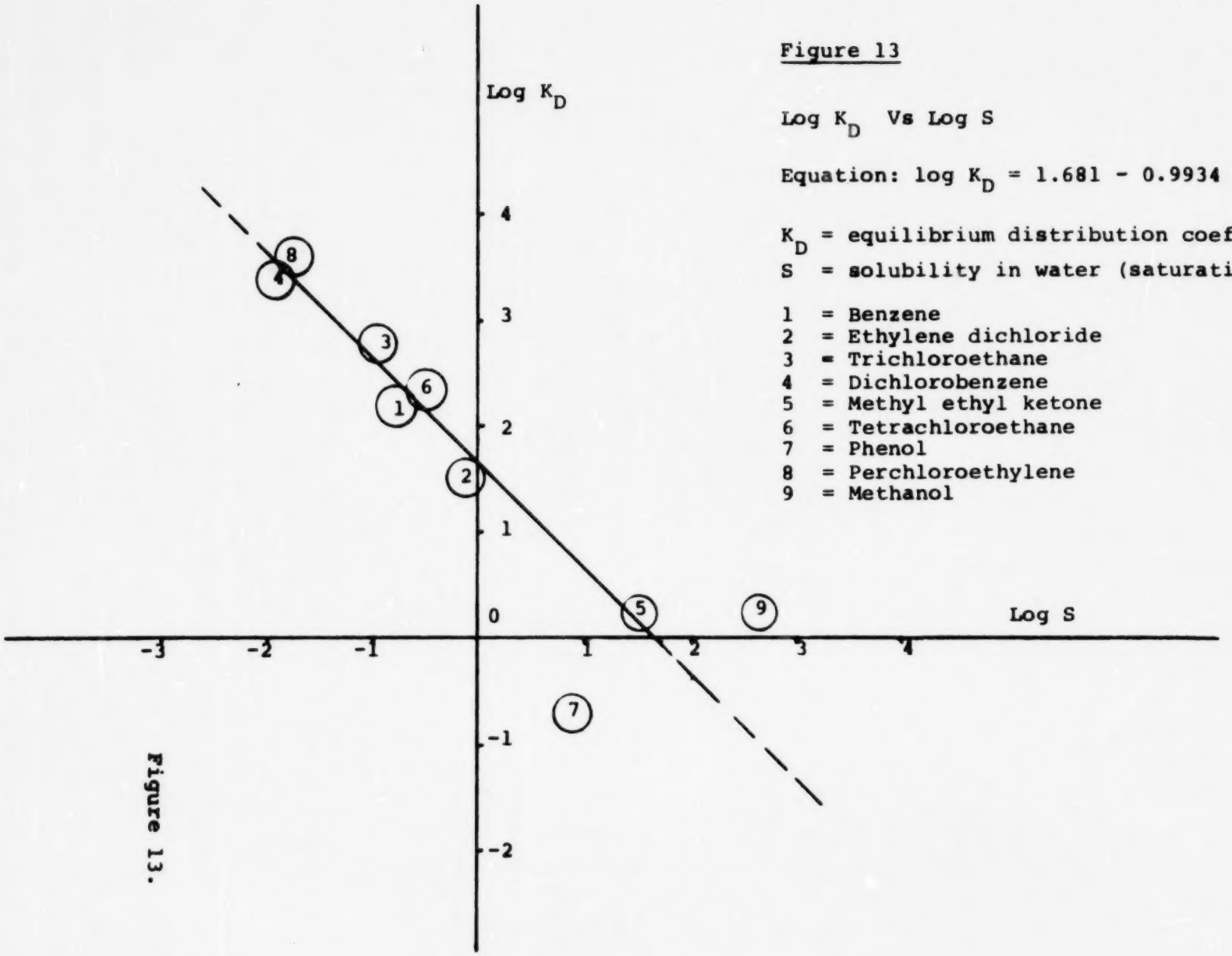


Figure 13.

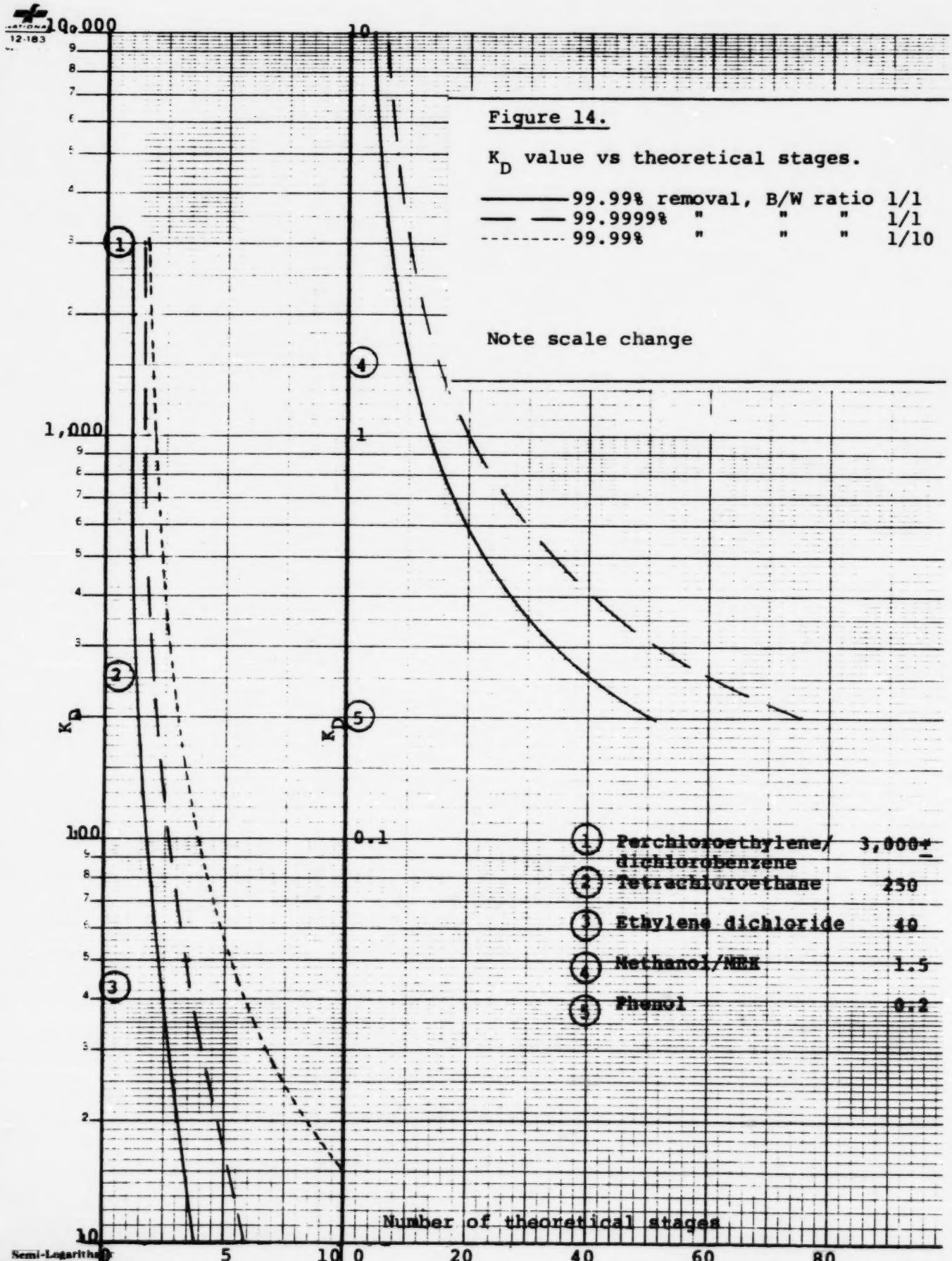
Figure 13

Log K_D Vs Log S

Equation: log K_D = 1.681 - 0.9934 log S

K_D = equilibrium distribution coefficient
S = solubility in water (saturation)

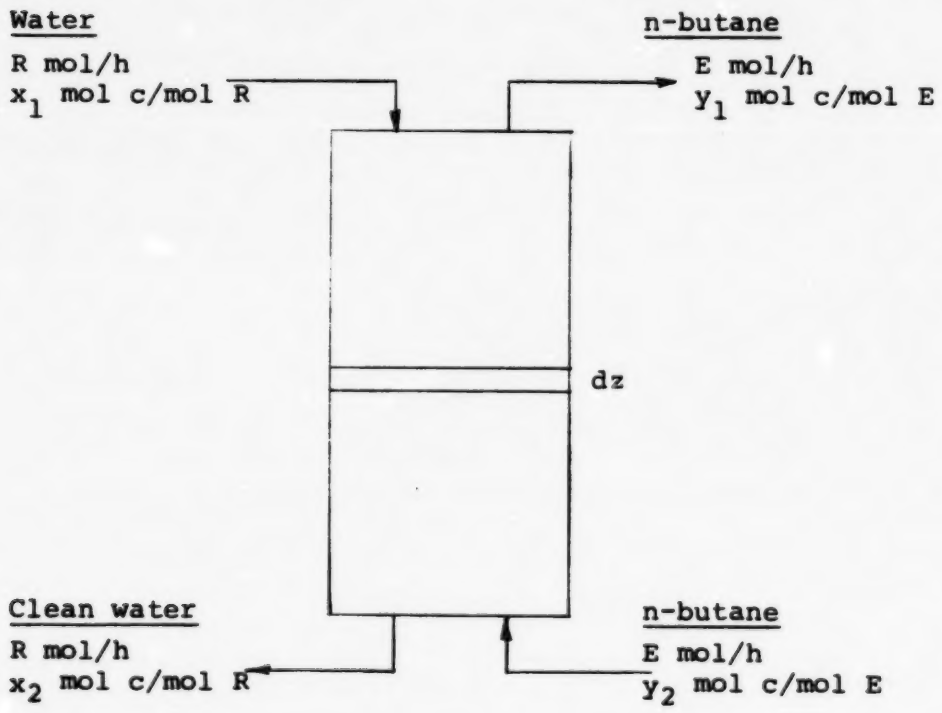
1 = Benzene
2 = Ethylene dichloride
3 = Trichloroethane
4 = Dichlorobenzene
5 = Methyl ethyl ketone
6 = Tetrachloroethane
7 = Phenol
8 = Perchloroethylene
9 = Methanol


10,000
1,000
100
10
1
0.1
K_D
K_D
1
2
3
4
5
0
5
10
0
20
40
60
80
Number of theoretical stages
Semi-Logarithmic
3 Cycles x 10 to the inch
Figure 14.
K_D value vs theoretical stages.
99.99% removal, B/W ratio 1/1
99.9999% " " " 1/1
99.99% " " " 1/10
Note scale change
1 Perchloroethylene/ dichlorobenzene 3,000+
2 Tetrachloroethane 250
3 Ethylene dichloride 40
4 Methanol/MEK 1.5
5 Phenol 0.2

6.8 Preliminary mathematical model

The purpose of this model is to allow us to calculate the size of extraction column needed to obtain a given contaminant removal based on a known water flow and inlet impurity level. The solutions are all dilute, as far as solvent extraction theory is concerned, and for each case only a single contaminant is considered.

Extraction is assumed to be carried out in a single countercurrent column as shown in the sketch below, where the contaminated water passes downwards through the column while n-butane passes upwards.



c = contaminant (e.g. ethylene dichloride)
x_1 = mol fraction of c in water entering column
x_2 = mol fraction of c in water leaving column
y_1 = mol fraction of c in butane leaving column
y_2 = mol fraction of c in butane entering column

For the case of dilute solutions, and for simple equilibria which can be represented by a distribution coefficient, the required height of the column is given by:

$$Z = HTU_{OR} * \ln \frac{[(x_1 - y_2/m)/(x_2 - y_2/m) * (1 - R/ME) + R/ME]}{1 - R/ME} \quad (1))$$

In equation (1) the first term is the height of a transfer unit (HTU_{OR}), and is a function of the type of contacting column and the fluid mechanics. The second term is the number of transfer units required (NTU_{OR}) based on the extraction required, the flowrates of water and n-butane, and the distribution coefficient (m).

$$NTU_{OR} = \ln [(x_1 - y_2/m)/(x_2 - y_2/m) * (1 - R/ME) + R/ME] \quad (2)$$

Here:
- R = mol/h of water
- E = mol/h of butane
- m = distribution coefficient (K_D)
- x_1 = mol fraction contaminant in inlet water
- x_2 = mol fraction contaminant in outlet water
- y_1 = mol fraction contaminant in inlet butane
- y_2 = mol fraction contaminant in outlet butane

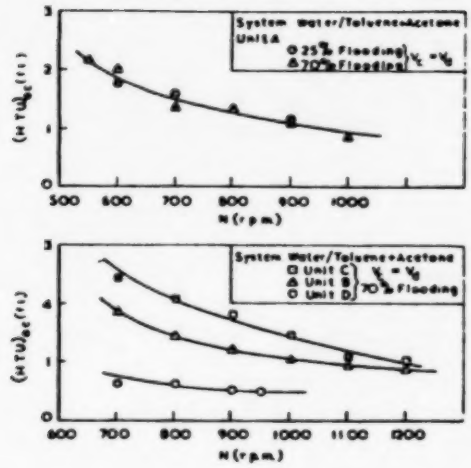
For a packed column of 13 mm saddles the height of a transfer unit may be calculated from:

$$HTU_{OR} = 0.95 + 2.72*(L_c/L_d)$$

Where:
- HTU_{OR} is in feet
- L_c is lb.mol/h of continuous phase
- L_d is lb.mol.h of dispersed phase

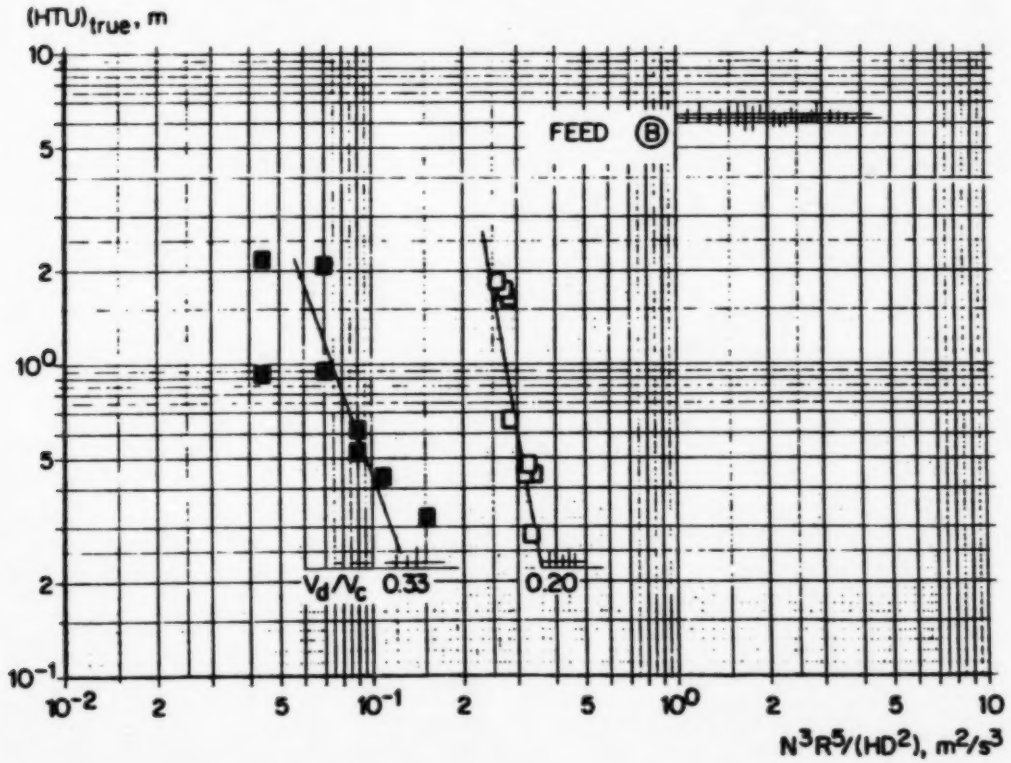
Clearly, if $L_c >> L_d$ the value of HTU_{OR} for a packed column will be large, making a packed column impractical for butane stripping. We have therefore discounted the use of packed columns for this application. For example, for a B/W ratio of 1/5, $(L_c/L_d) = 27.8$, and HTU_{OR} will be over 75 feet.

For rotating disc contactor (RDC) columns some very complicated correlations are available. These require calculation of dispersed phase hold-up, droplet rise velocity and column geometry factors. Such correlations are too complex and fraught with uncertainties for use in these preliminary calculations. Instead, the raw experimental data reported in the literature will be used. Figures 15 and 16 show such typical data.



Above. Figure 15. Typical plots showing variations in overall HTU of an RDC with rotor speed (from Logsdail et al, 1957).

Below. Figure 16. Plots of HTU versus factors incorporating rotor speed and rotor diameter (from Bruin et al, 1974).



From the data in Figures 15 and 16, it is clear that at reasonably high rotation speeds HTU_{OR} values of about 1 to 1.5 feet can be achieved. For calculation purposes an HTU_{OR} of 1.5 feet is assumed for the RDC.

Calculations were done for three typical contaminants:

ethylene dichloride	m (K_D)	= 45
1,1,2,2-tetrachloroethane	m	= 250
1,1,1-trichloroethane	m	= 587

These three species cover a range of K_D values for which a single solvent process could be feasible. K_D values greater than 587 will, of course, make the required column even smaller.

Two values of the butane/water ratio were selected - volumetric ratios of 1/5 and 1/2.5. Results are given in Tables 3 - 8.

Table 3. Column height versus contaminant removal.
Contaminant: ethylene dichloride
B/W ratio : 1/5

Contaminant removal (%)	NTU_{OR}	RDC column height ft	m
98.0	7.545	11.3	3.45
99.0	9.236	13.8	4.21
99.8	13.22	19.8	6.04
99.9	14.94	22.5	6.86
99.98	18.94	28.5	8.69

Table 4. Column height versus contaminant removal.
Contaminant: ethylene dichloride
B/W ratio : 1/2.5

Contaminant removal (%)	NTU_{OR}	RDC column height ft	m
98.0	5.087	7.6	2.32
99.0	6.071	9.1	2.77
99.8	8.363	12.5	3.81
99.9	9.351	14.0	4.27
99.98	11.64	17.5	5.34

Table 5. Column height versus contaminant removal.
Contaminant: 1,1,2,2-tetrachloroethane
B/W ratio : 1/5

Contaminant removal (%)	NTU_{OR}	RDC column height ft	m
98.0	6.838	10.3	3.14
99.0	7.614	11.4	3.48
99.8	9.418	14.1	4.30
99.9	10.19	15.3	4.66
99.98	11.99	18.0	5.49

Table 6. Column height versus contaminant removal.
Contaminant: 1,1,2,2-tetrachloroethane
B/W ratio : 1/2.5

Contaminant removal (%)	NTU_{OR}	RDC column height ft	m
98.0	4.077	6.12	1.87
99.0	4.809	7.2	2.2
99.8	6.510	9.8	2.99
99.9	7.241	10.9	3.32
99.98	8.944	13.4	4.06

Table 7. Column height versus contaminant removal.
Contaminant: 1,1,1-trichloroethane
B/W ratio : 1/5

Contaminant removal (%)	NTU_{OR}	RDC column height ft	m
98.0	4.05	6.1	1.86
99.0	4.78	7.2	2.20
99.8	6.46	9.7	2.96
99.9	7.19	10.8	3.29
99.98	8.88	13.3	4.05

Table 8. Column height versus contaminant removal.
Contaminant: 1,1,1-trichloroethane
B/W ratio : 1/2.5

Contaminant removal (%)	NTU_{OR}	RDC column height ft	m
98.0	3.98	6.0	1.83
99.0	4.69	7.0	2.13
99.8	6.34	9.5	2.90
99.9	7.05	10.6	3.23
99.98	8.69	13.0	3.96

The above tabulations refer to RDC columns which have been referenced in the literature. More effective columns may be possible for the specific butane/water case; thus allowing shorter columns. This is an area that requires R & D effort.

7. MASS AND ENERGY BALANCES

7.1 Introduction

The mass and energy balances for the butane stripping process are based on the following assumed conditions:

- ambient temperature 20°C
- water flow rate 1,000 l/h
- butane flow rate 200 l/h
- datum temperature 0°C
- reflux rate 133 kg/h (this is required for the methanol case but should be much less for other cases; for example, it needs to be only 13 kg/h for a butane/benzene fractionation)

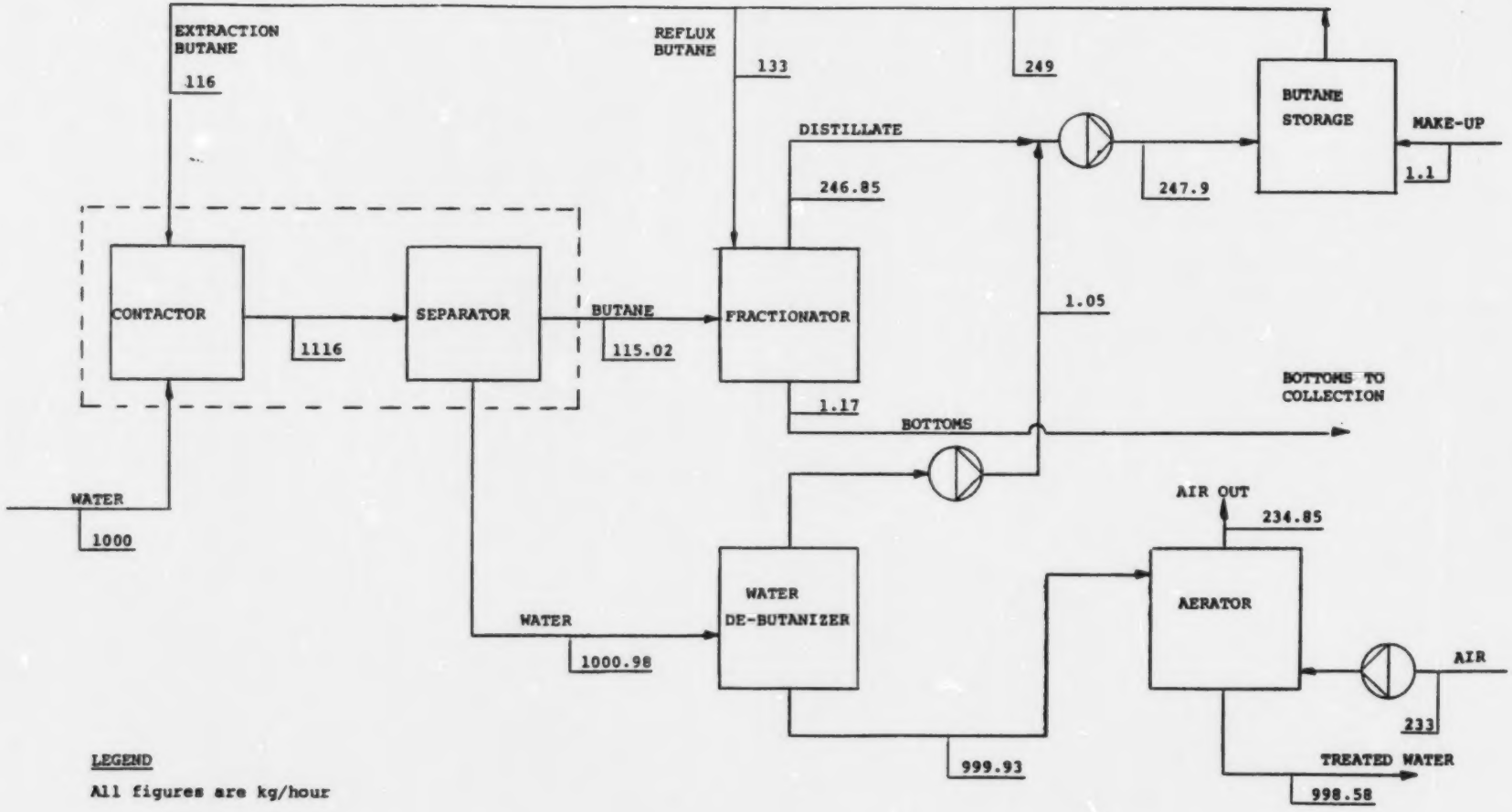
7.2 Mass balance

The mass balance for the process is given on Figure 17, and is tabulated below. The diagram is self-explanatory, and no extensive explanation is necessary. Butane losses from the system (requiring replacement with purchased butane) are calculated at 0.10 kg/h, with 90% of this amount being stripped from the water in an air stripping column prior to discharge to the receiving water. Note that due to the small amounts involved, some of the figures are approximated.

Mass balance

Inputs(kg/hour)		Outputs(kg/hour)	
Water	1,000	Water to disposal	998.7
Butane	116	Water in butane	negl.
Reflux butane	133	Butane to storage	247.9
Air	233	Butane in bottoms	1.1
		Butane in water	negl.
		Butane in air	0.10 max.
		Water in air	1.2
		Air	233
Total	1,482		1,482

Net removal from the circulating system includes 1.1 kg/h butane in the fractionator bottoms (recoverable) and 0.10 kg/h butane in the water to the air stripping column (lost). Optimisation of the fractionation column design and operation should enable a bottom product to be produced containing much less butane than assumed for this mass balance and economic analysis.



MASS BALANCE FOR BUTANE STRIPPING PROCESS AT MAXIMUM REFLUX RATE (B/W ratio 1/5)

FIGURE 17

7.3 Energy balance

The energy balance for the process is shown on Figure 18. As with the mass balance, it is self-explanatory. Principal energy inputs to the system are:

- contactor agitator driver	5 HP electric motor
- water supply pump	1/2 HP electric motor
- main butane compressor	3 HP electric motor
- secondary compressor/vac	1/2 HP electric motor
- air blower	1/4 HP electric motor
- fractionation column	76,200kJ/h

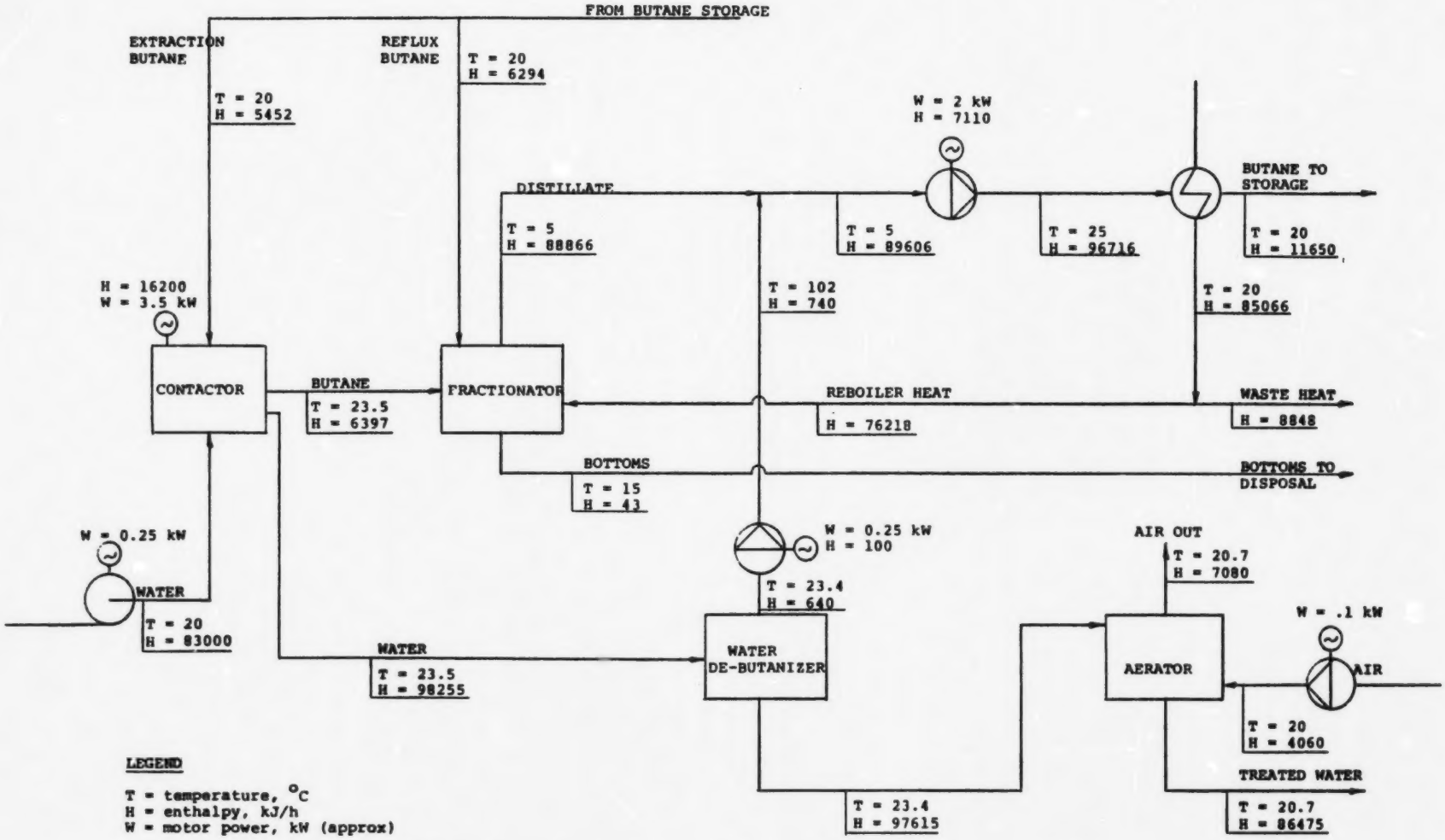
The total electrical power demand (10 HP) can be supplied with a small gas or diesel generator. The heat requirement for the fractionation column can be provided by the heat of compression of the main butane compressor or from exhaust and/or cooling system heat from the generator. The generator engine could possibly be used to burn vapour from the fractionation column bottom product.

Based on the electric driver requirements (10 HP) the total system energy requirement is as follows:

Total power demand	10 HP
Thermal equivalent	25,450 BTU/h
Engine efficiency (estimate)	20%
Engine heat input	127,250 BTU/h
Available waste heat (50%)	50,900 BTU/h
Cost of diesel fuel	$0.30/l
Heating value of diesel fuel	35,000 BTU/l
Diesel fuel consumption	3.64 l/h
Diesel fuel cost	$1.09/h

The additional heating loads to the system can be taken from the heat rejected by the diesel generator.

Butane consumption by the system will be 0.10 kg/h. The butane rejected from the system in the fractionator bottom product (1 kg/h) is recoverable by further fractionation or by utilization as a fuel. Therefore, from the standpoint of operating cost, this butane "loss" should not be counted as a true cost to the process. However, for the sake of deriving comparative costs, the replacement of butane is assumed to be 0.10 kg/h (average case) or 1.1 kg/h (worst case).



ENERGY BALANCE FOR BUTANE STRIPPING PROCESS AT MAXIMUM REFLUX RATE (B/W ratio 1/5)

FIGURE 18

	Average case	Worst case
Butane loss	0.10 kg/h	1.10 kg/h
Butane cost	$0.40/kg	$0.40/kg
Operating cost	$0.04/h	$0.44/h

Total operating cost = ($1.09 + $0.04)/hour = $1.13/h (Average)
= ($1.09 + $0.44)/hour = $1.53/h (Worst)
Water treatment cost = $1.13 (Ave) to $1.53 (Worst) per 1,000 litres.

The above costs are negligible compared with the cost of mobilization, transport and operating personnel of any stripping system. We estimate that a butane stripper would have a crew of two persons, with a real operating labour cost of about $80/h (wages, benefits and overhead). A steam stripper would have a similar crew requirement. Operation could be entirely automatic, via computer control, or could be manual. Regardless of the mode of operation, the operating cost will be about the same.

The butane stripper should be a very simple system to operate. We have found in our short program that liquid butane is easy to handle. It separates quickly and cleanly from the water layer with no indication of any emulsion formation. It distills off the organic contaminant quite easily, and heat input needs to be only low level heat, since the process operates at low temperature. Liquid butane has a relatively low latent heat of vapourization (92 cal/g @ -0.5°C) so phase changes can be accomplished with minimal energy input. This compares with the high heat of vapourization of water (540 cal/g @ 100°C) requiring high quality energy input.

8. COMPARISON WITH STEAM STRIPPING.

Liquid-liquid extraction, with a volatile solvent like butane, differs considerably from steam stripping, and has many theoretical advantages. The principal differences are:

1. In steam stripping the entire waste water stream must be heated almost to its atmospheric boiling point, thereby requiring a substantial energy input (Earhart et al, 1976). Energy must be supplied to the process at a high temperature to generate the steam required in the process. On condensing, much of the heat input is lost to the waste water stream or to condenser water.

   In contrast, the butane stripping process requires relatively low energy input, this being principally to the butane compressors, to the mixer and to the process pumps. The compressor energy is recovered by utilizing the heat of compression in the process. The low boiling point of butane (-0.5°C) means that low temperature energy can be used to provide much of the heat of evaporation for the butane. A complete mass and energy balance for the butane stripping process is contained in Section 7 of this report.

2. With all but the lowest molecular weight organic compounds, an azeotrope will occur at some point in the water/pollutant binary system, limiting the purity of the concentrated organic product (Earhart et al, 1976). In practice, steam stripping may produce a persistent emulsion with many organic compounds which is difficult to separate (Earhart et al, 1976). It will be a formidable task to completely isolate pure water and pure organic compound from the process streams.

   Butane stripping may also form an azeotrope with some organics, although we are not aware of any at present, and the difference in boiling point between butane (-0.5°C) and most organic pollutants (mainly liquids at standard conditions) will allow fractionation to give relatively pure butane and organic fractions. Butane stripping will give a residue which is easily incinerated and will not give an aqueous sludge/emulsion for subsequent disposal. The residue from butane stripping may be amenable to further concentration to recover additional pure butane and concentrated pollutant.

3. For corrosive organics steam stripping, and the elevation in temperature required, will aggravate any corrosion problems. This will apply to the steam stripping plant and also to the storage and handling of stripper residues. Butane stripping will be affected by any corrosivity of the organic component, but will not worsen the situation by introducing water or by raising the system temperature significantly.

4. Steam stripping may cover a broader range of chemical pollutants than butane stripping and may recover some chemical pollutants that butane stripping cannot recover efficiently. Butane stripping may be a more specialized process, limited to a narrower range of non-polar

organics. However, in this area butane stripping is expected to outperform steam stripping substantially as regards extraction and energy efficiency and cost effectiveness. Since many organic pollutants of greatest concern are non-polar and hydrophobic organics, butane stripping will have wide applicability. For polar organics, a dual solvent system offers the opportunity to achieve 99.99+% removal efficiency. This topic is discussed later in this report.

It should be noted that steam stripping (or steam distillation) is generally used to distill organics having high boiling points and low vapour pressures. It is aimed at increasing the partial pressure of the organic component in the vapour and increasing its volatility. Many organic pollutants are volatile and may not respond well to partial pressure manipulation. Since butane stripping is not controlled by partial pressure, it will extract both low and high boiling point organics equally well.

5. In pollutant clean-up, time is of the essence. It is important that any stripping system be brought to site quickly and put in operation as soon as possible. In this regard, steam stripping is clearly inferior to butane stripping.

   Steam stripping requires that the steam generator and all process equipment be kept drained and protected against corrosion during inactive periods. In winter, freezing is a major concern and equipment storage and transport must be treated with caution. It may be necessary to store the equipment indoors in a heated area to prevent freezing damage. The result of these restrictions is that the steam stripper will take longer to put into storage, longer to get out of storage, longer to get on site, and longer to start-up on-site than a butane stripper.

   Butane stripping utilizes a liquid that boils at -0.5°C and freezes at -138.6°C. It can be stored outside under any conditions without fear of freezing. Most energy input to the system will be from a built-in diesel generator driving the butane compressors, blower and system pumps, with possibly a small butane burner as back-up heating to the butane evaporator. The stripper can be designed as a sealed system ready for immediate start-up. We estimate the stripper could be mobilized faster than its operating crew can be assembled, and could be in operation on-site within an hour of arrival.

6. For certain organic chemicals, butane stripping gives such remarkable clean-up results that it must be considered as the preferred process. Reductions of 97.5 - 99.8% of the original concentrations were demonstrated for 6 out of 9 organic pollutants tested using a single butane extraction stage. With 3-4 stages of extraction, as would be incorporated in a commercial system, the concentration in water should be reduceable by 99.99% for a large number of organic pollutants. This level of extraction efficiency was in fact demonstrated in pesticide removal (Reynolds, 1983) using hexane and pentane as the solvents.

Iso-butane was used to extract benzene, toluene and xylene from ethylene quench water with efficiencies of 97%, 96.3% and >97% respectively in a single stage extraction (Earhart et al, 1977). In the same test, butane was ineffective in extracting phenol (6% extraction) and a dual solvent process is recommended by the authors to give 99% removal of phenolics.

One possibility for the removal of phenol from water is to utilize a selective solvent (e.g. n-butyl acetate) that will extract the phenol efficiently (K_D = 65) then to remove residual butyl acetate from the water using butane (K_D = 107) (Earhart et al, 1977). In such a two-step process, highly effective phenol removal is possible in a small number of extraction stages (theoretically, 99.9% removal in two butyl acetate stages, followed by two butane stages - a total of four stages). Although a theoretical consideration, this type of approach may allow butane stripping to become a universally applicable technology.

7. The butane stripper has the potential for explosion and/or fire because of the flammability of butane gas. It will therefore require more careful engineering, use of explosion-proof electrical equipment, and stringent safety precautions during operation. Since these conditions prevail in most petroleum refineries and in gas plants, we do not view this as a major problem.

   Steam stripping does not have these disadvantages, except that the fuel supply to the steam boiler will require care in handling. It does carry the risk of scalds to operating personnel resulting from a steam leak.

9. CONCLUSIONS AND RECOMMENDATIONS

It has been demonstrated convincingly that the butane stripping process is an extremely effective method for extracting organic pollutants from water. Removal efficiencies as high as 99.8% were recorded using a simple, single-stage apparatus in the laboratory. K_D values derived from our test work agree with values reported in the literature.

The results from our laboratory tests demonstrate that butane stripping is a very promising technique for decontaminating waters polluted with organic chemicals, although further work is required before the process can be called a proven technology. Much of our information on the process is theoretical or calculated based on the preliminary data from our tests. However, the data we obtained on pollutant extraction are so remarkable that we have no doubt that a practical system can be developed to recover 99.99+% of many organic pollutants from water. The extraction efficiencies obtained are believed to be significantly better than could be achieved with any reasonable alternative.

The program should be continued to develop more information on the butane stripping process and to develop the following specifics:

1. Undertake an expanded series of single-stage extraction tests on a wide range of organic chemicals to obtain K_D values for as many organics as may be practical. The tests would utilize the existing test apparatus and could cover families of organics (i.e. homologues), providing data on K_D values that would allow extraction performance to be predicted. This would also include the further development of the preliminary correlation between K_D and Solubility discovered during our first tests.

2. Examine butane removal/recovery from water, and particularly the process conditions that allow high efficiency extraction to be achieved. Examine column packing materials such as expanded polystyrene for their suitability and practicality. Investigate the effect of rapid pressure drop, butane evacuation and residence time on butane removal from water.

3. Develop a design for a multi-stage contactor that will be effective, rugged and compact, suitable for mounting on a truck or trailer. Construct and test a suitable contactor, with an efficient butane/water separator.

4. Develop a pilot-scale extraction process to treat a water flow up to 5 l/h on a continuous basis, with extraction in a multi-stage contactor. This will incorporate the contactor from 3 above, as well as the fractionation column, heat exchangers, compressors and pumps needed to make the apparatus an operable system.

5. Perform test runs on the assembled pilot-plant on a range of organic contaminants specified by Environment Canada. Prove conclusively that the concept and its practical application are feasible for a field installation. Confirm that the mass and energy balances predicted for the system are reasonable.

Following the test program with the pilot-plant, a decision would be made on whether to proceed further with the project or to abandon it. Assuming that the pilot-plant testing was successful, and a go decision was made, we would proceed to the next phase:

6. Design a butane stripper for field use mounted on a truck or trailer, complete with all necessary auxiliaries,including an engine-generator unit, controls, switchgear, etc. System specification to be agreed with Environment Canada personnel. Acquire the system components and construct a prototype butane stripper from the approved design. Take the unit into the field and perform field testing as required by Environment Canada. Make modifications and/or improvements to the pilot-plant during the initial testing period.

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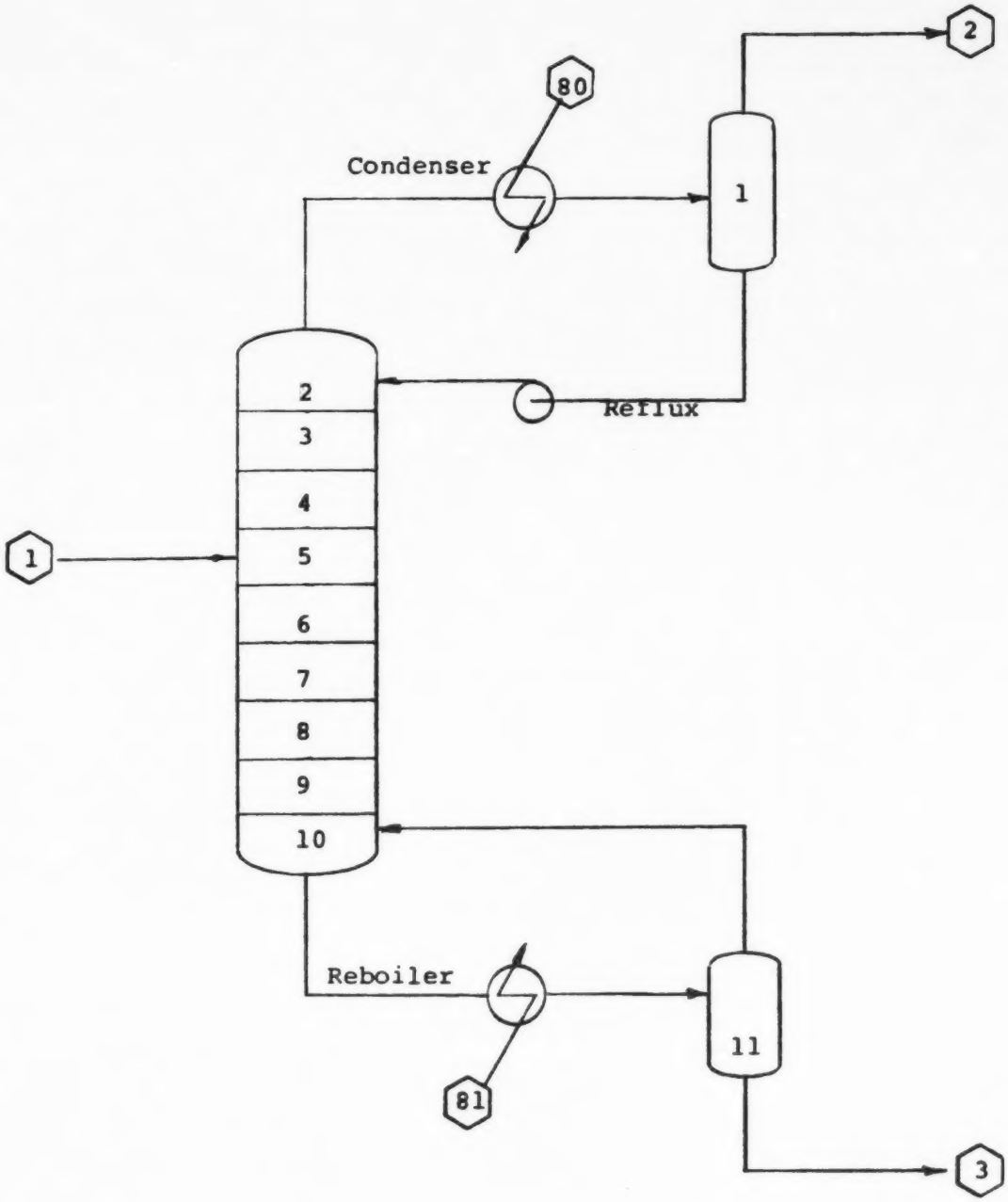
APPENDICES

APPENDIX 1

HYSIM computer simulation

A computer simulation run on two feed mixtures; butane/methanol in the ratio 99.5/0.5 and butane/benzene in the ratio 99.5/0.5. Required butane output quality is 99.999%. Fractionation column used in the trial had 11 stages, as shown on the drawing.



Schematic of 11 stage fractionation column evaluated in the HYSIM process simulator. Feed on to the 5th tray, reboiler is a stage, as is distillate receiver. Points shown thus ⬡ identified in the print-out.

Stream Name		1	2	3	80
Vapour frac.		0.0000	1.0000	0.0000	2.0000*
Temperature	F	74.3000*	41.1775*	54.8626*	32.0000*
Pressure	Psia	34.7000*	18.0000*	20.0000*	0.0000*
Molar Flow	Lbmole/hr	3.6600*	3.5687*	0.0913*	0.0000*
Mass Flow	Lb/hr	212.2563	207.4247	4.8316	0.0000
LiqVol Flow	Barrel/Day	24.8511	24.3032	0.5479	0.0000
Enthalpy	Btu/hr	-4037.9695	26447.4766	-328.3671	-23773.7930*
Density	Lb/ft3	35.4575	0.2032	38.3880	0.0000
Mole Wt.		57.9936	58.1237	52.9076	0.0000
Spec. Heat	Btu/lbmo-F	33.2855	22.3824	30.5121	0.0000
Therm Cond	Btu/h-ft-F	0.0621	0.0086	0.0934	---
Viscosity	Cp	0.1595	0.0067	0.2456	---
Z Factor		0.0099	0.9580	0.0050	---
Sur Tension	Dyne/cm	12.1849	---	16.2419	---
Std Density	Lb/ft3	36.0207	---	38.1847	---
n-Butane		0.9950*	1.0000*	0.8000*	0.0000*
Methanol		0.0050*	0.0000*	0.2000*	0.0000*

Stream Name		81
Vapour frac.		2.0000*
Temperature	F	32.0000*
Pressure	Psia	0.0000*
Molar Flow	Lbmole/hr	0.0000*
Mass Flow	Lb/hr	0.0000
LiqVol Flow	Barrel/Day	0.0000
Enthalpy	Btu/hr	53916.3125*
Density	Lb/ft3	0.0000
Mole Wt.		0.0000
Spec. Heat	Btu/lbmo-F	0.0000
Therm Cond	Btu/h-ft-F	---
Viscosity	Cp	---
Z Factor		---
Sur Tension	Dyne/cm	---
Std Density	Lb/ft3	---
n-Butane		0.0000*
Methanol		0.0000*

HYSIM Hydrocarbon simulation

Trial on a butane/methanol feed

Column conditions, inputs and outputs

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:26:34
Column Name A:BUTMETH

Number of Stages	11	
Stage 1 Pressure	18.000	Psia
Stage 11 Pressure	20.000	Psia
Stage 1 Temperature	41.000.	F
Stage 11 Temperature	70.000	F

Feed stream 1 enters on stage 5
Overhead Vapour Estimated Flow 3.5000 Lbmole/hr
Estimated top stage reflux ratio is 3.00000
Side Exchanger on stage 11
Side Exchanger on stage 1 is energy stream 80
Overhead vapour product goes to stream 2
Bottom liquid product goes to stream 3
Condenser energy goes to stream 80
Reboiler energy goes to stream 81

Specifications

1: Molar ratio of n-Butane divided by Methanol in the stage 11 Liquid is to be	4.0000	
2: Mole Fraction of Methanol in the stage 1 Vapour is to be	0.0000	

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:27:17
Column Name A:BUTMETH

***** COMPUTED RESULTS *****

Reflux Ratio 0.6960

Feed Conditions

Feed Stream Number	1	
Enters on stage	5	
Liquid Fraction	1.0000	
Temperature	74.30	F
Pressure	34.70	Psia
Flowrate	3.66	Lbmole/hr
Enthalpy	-4038.	Btu/hr
n-Butane	3.6417	Lbmole/hr
Methanol	0.0183	Lbmole/hr

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:27:18
Column Name A:BUTMETH

*** Stage Variables ***

Stage	Press Psia	Temp Deg F	Molar Flow Rates Liquid	Vapour	Feed	Draws	Heat Duty MM Btu/Hour
1	18.00	41.18	2.5			3.6 V	-0.0238
2	18.20	41.73	2.5	6.1			
3	18.40	42.30	2.5	6.0			
4	18.60	42.88	2.5	6.0			
5	18.80	43.52	5.7	6.0	3.7		
6	19.00	44.05	5.8	5.7			
7	19.20	44.58	5.8	5.7			
8	19.40	45.13	5.8	5.7			
9	19.60	45.83	5.7	5.7			
10	19.80	47.41	5.6	5.6			
11	20.00	54.86		5.5		0.1 L	0.0539

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:27:19
Column Name A:BUTMETH

*** Products ***

	Vapour from Stage 1		Liquid from Stage 11	
	Lb Mol/Hour	Mol Frc	Lb Mol/Hour	Mol Frc
n-Butane	3.569	0.99999	0.073	0.80000
Methanol	0.000	0.00001	0.018	0.20000
Total	3.569		0.091	

Hyprotech's Process Simulator HYSIM Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:27:27
Column Name A:BUTMETH

** PHYSICAL PROPERTIES **

VAPOUR LEAVING FROM EACH STAGE

STAGE	MOL WT	FLOW LB/HOUR	M ACFH	DENSITY LB/FT3	REDUCED TEMP	REDUCED PRESS
1	58.124	207.	1.021	0.20319	0.65439	0.03269
2	58.123	352.	1.714	0.20529	0.65512	0.03305
3	58.122	352.	1.696	0.20739	0.65584	0.03341
4	58.118	351.	1.678	0.20946	0.65658	0.03377
5	58.108	351.	1.660	0.21148	0.65732	0.03412
6	58.108	329.	1.539	0.21358	0.65806	0.03448
7	58.107	329.	1.525	0.21568	0.65876	0.03485
8	58.105	329.	1.511	0.21775	0.65946	0.03521
9	58.088	329.	1.497	0.21971	0.66023	0.03555
10	57.985	327.	1.482	0.22088	0.66182	0.03578
11	57.343	315.	1.453	0.21716	0.66816	0.03532

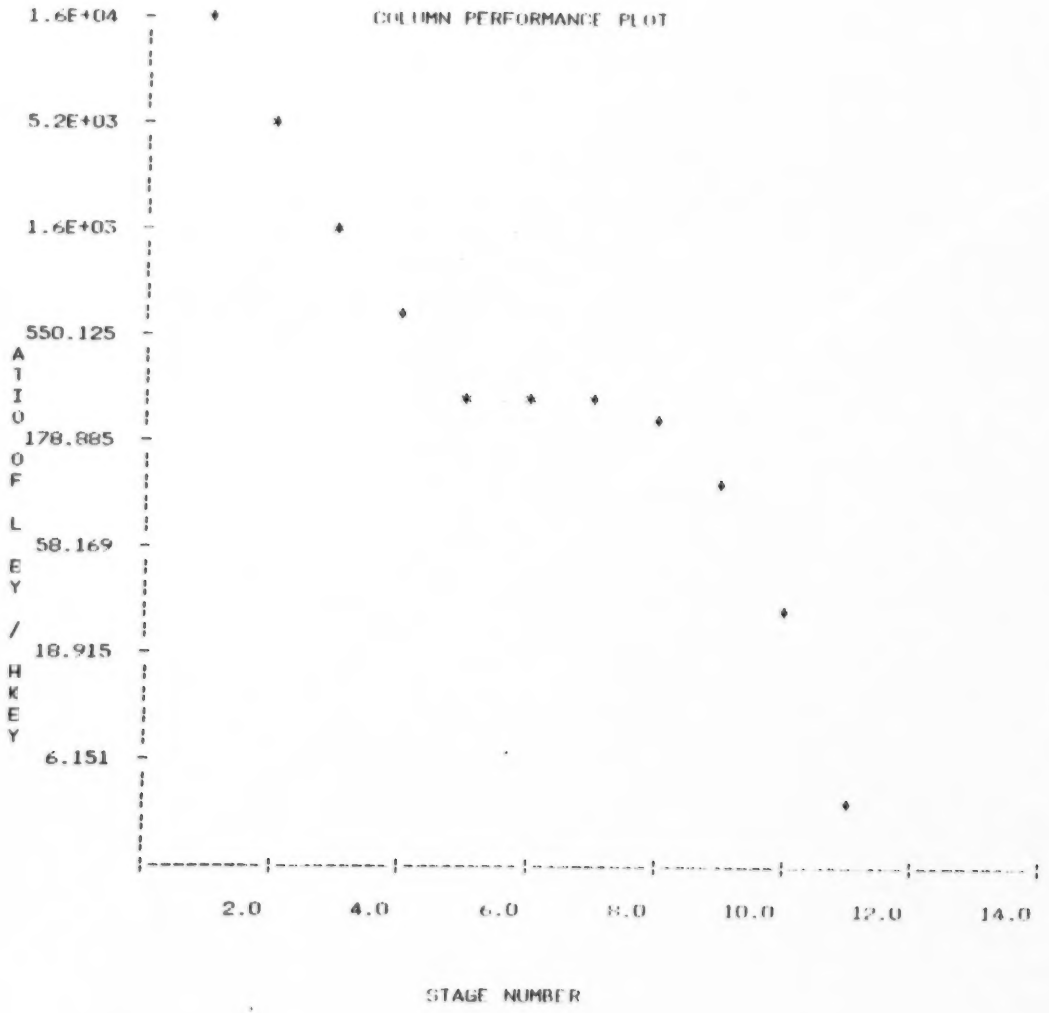
LIQUID LEAVING FROM EACH STAGE

STAGE	MOL WT	FLOW LB/HOUR	USGPM	DENSITY LB/FT3	REDUCED TEMP	REDUCED PRESS
1	58.122	144.	0.5	36.69042	0.65439	0.03269
2	58.119	144.	0.5	36.67091	0.65509	0.03305
3	58.109	144.	0.5	36.65348	0.65578	0.03340
4	58.086	144.	0.5	36.64111	0.65642	0.03373
5	58.025	333.	1.1	36.64131	0.65694	0.03402
6	58.025	334.	1.1	36.62131	0.65763	0.03438
7	58.022	334.	1.1	36.60221	0.65831	0.03474
8	58.006	334.	1.1	36.58793	0.65895	0.03508
9	57.904	332.	1.1	36.60268	0.65933	0.03531
10	57.271	320.	1.1	36.80026	0.65812	0.03488
11	52.908	5.	0.0	38.38933	0.64573	0.03056

STAGE	LKEY FRAC	HKEY FRAC	RATIO
1	0.99994	0.00006	15725.78711
2	0.99980	0.00020	4943.01611
3	0.99944	0.00056	1788.93921
4	0.99853	0.00147	679.72449
5	0.99622	0.00378	263.44666
6	0.99620	0.00380	262.22333
7	0.99610	0.00390	255.30754
8	0.99547	0.00453	219.69083
9	0.99158	0.00842	117.71936
10	0.96729	0.03271	29.56921
11	0.80000	0.20000	3.99997

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:30:35
Column Name A:BUTMETH



Stream Name		1	2	3	80
Vapour frac.		0.0000	1.0000	0.0000	2.0000*
Temperature	F	74.3000*	41.1819*	56.8867*	32.0000*
Pressure	Psia	34.7000*	18.0000*	20.0000*	0.0000*
Molar Flow	Lbmole/hr	3.6600*	3.5687*	0.0913*	0.0000*
Mass Flow	Lb/hr	213.0993	207.4254	5.6739	0.0000
LiqVol Flow	Barrel/Day	24.9113	24.3032	0.6081	0.0000
Enthalpy	Btu/hr	-3993.3738	26447.7676	-272.1728	-5875.2725*
Density	Lb/ft3	35.4997	0.2032	39.8331	0.0000
Mole Wt.		58.2239	58.1242	62.1205	0.0000
Spec. Heat	Btu/lbmo-F	33.2990	22.3827	31.1144	0.0000
Therm Cond	Btu/h-ft-F	0.0617	0.0086	0.0694	---
Viscosity	Cp	0.1516	0.0067	0.2111	---
Z Factor		0.0099	0.9580	0.0056	---
Sur Tension	Dyne/cm	12.1956	---	16.1413	---
Std Density	Lb/ft3	36.0620	---	39.7167	---
n-Butane		0.9950*	1.0000*	0.8000*	0.0000*
Benzene		0.0050*	0.0000*	0.2000*	0.0000*

Stream Name		81
Vapour frac.		2.0000*
Temperature	F	32.0000*
Pressure	Psia	0.0000*
Molar Flow	Lbmole/hr	0.0000*
Mass Flow	Lb/hr	0.0000
LiqVol Flow	Barrel/Day	0.0000
Enthalpy	Btu/hr	36045.3828*
Density	Lb/ft3	0.0000
Mole Wt.		0.0000
Spec. Heat	Btu/lbmo-F	0.0000
Therm Cond	Btu/h-ft-F	---
Viscosity	Cp	---
Z Factor		---
Sur Tension	Dyne/cm	---
Std Density	Lb/ft3	---
n-Butane		0.0000*
Benzene		0.0000*

HYSIM Hydrocarbon simulation

Trial on a butane/benzene feed

Column conditions, inputs and outputs

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:35:40

Number of Stages	11	
Stage 1 Pressure	18.000	Psia
Stage 11 Pressure	20.000	Psia
Stage 1 Temperature	41.000	F
Stage 11 Temperature	70.000	F

Feed stream 1 enters on stage 5
Overhead Vapour Estimated Flow 3.5000 Lbmole/hr
Estimated top stage reflux ratio is 3.00000
Side Exchanger on stage 11
Side Exchanger on stage 1 is energy stream 80
Overhead vapour product goes to stream 2
Bottom liquid product goes to stream 3
Condenser energy goes to stream 80.
Reboiler energy goes to stream 81

Specifications

1: Molar ratio of n-Butane
divided by

		Benzene
in the stage 11 Liquid is to be		4.0000

2: Mole Fraction of Benzene
in the stage 1 Vapour is to be 0.0000

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:35:41

***** COMPUTED RESULTS *****

Reflux Ratio 0.17110

Feed Conditions

Feed Stream Number	1	
Enters on stage	5	
Liquid Fraction	1.0000	
Temperature	74.30	F
Pressure	34.70	Psia
Flowrate	3.66	Lbmole/hr
Enthalpy	-3993.	Btu/hr
n-Butane	3.6417	Lbmole/hr
Benzene	0.0183	Lbmole/hr

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:35:42

*** Stage Variables ***

Stage	Press Psia	Temp Deg F	Molar Flow Rates Liquid	Vapour	Feed	Draws	Heat Duty MM Btu/Hour
1	18.00	41.18	0.6			3.6 V	-0.0059
2	18.20	41.75	0.6	4.2			
3	18.40	42.32	0.6	4.2			
4	18.60	42.93	0.6	4.2			
5	18.80	43.60	3.9	4.2	3.7		
6	19.00	44.13	3.9	3.8			
7	19.20	44.66	3.9	3.8			
8	19.40	45.19	3.9	3.8			
9	19.60	45.76	3.9	3.8			
10	19.80	46.98	3.8	3.8			
11	20.00	56.89		3.7		0.1 L	0.0360

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

Case Name A:BUTCOL Date 1987/12/30 Time 15:35:57

*** Products ***

	Vapour from Stage 1		Liquid from Stage 11	
	Lb Mol/Hour	Mol Frc	Lb Mol/Hour	Mol Frc
n-Butane	3.569	0.99999	0.073	0.80003
Benzene	0.000	0.00001	0.018	0.19997
Total	3.569		0.091	

Hyprotech's Process Simulator HYSIM - Licensed to Westcoast Transmission Co

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** PHYSICAL PROPERTIES **

VAPOUR LEAVING FROM EACH STAGE

STAGE	MOL WT	FLOW LB/HOUR	M ACFH	DENSITY LB/FT3	REDUCED TEMP	REDUCED PRESS
1	58.124	207.	1.021	0.20319	0.65440	0.03269
2	58.125	243.	1.183	0.20529	0.65513	0.03305
3	58.125	243.	1.170	0.20739	0.65584	0.03341
4	58.127	242.	1.157	0.20947	0.65665	0.03378
5	58.131	242.	1.145	0.21153	0.65748	0.03414
6	58.131	220.	1.028	0.21363	0.65818	0.03450
7	58.131	220.	1.019	0.21573	0.65887	0.03486
8	58.131	220.	1.010	0.21783	0.65956	0.03523
9	58.133	220.	1.001	0.21991	0.66028	0.03559
10	58.153	220.	0.992	0.22174	0.66167	0.03594
11	58.443	215.	0.977	0.22042	0.67147	0.03615

LIQUID LEAVING FROM EACH STAGE

STAGE	MOL WT	FLOW LB/HOUR	USGPM	DENSITY LB/FT3	REDUCED TEMP	REDUCED PRESS
1	58.127	35.	0.1	36.67234	0.65437	0.03269
2	58.133	35.	0.1	36.67056	0.65504	0.03305
3	58.145	35.	0.1	36.66847	0.65567	0.03340
4	58.172	35.	0.1	36.66693	0.65618	0.03375
5	58.225	225.	0.8	36.69254	0.65649	0.03409
6	58.225	225.	0.8	36.67239	0.65718	0.03445
7	58.225	226.	0.8	36.65245	0.65787	0.03482
8	58.227	226.	0.8	36.63375	0.65854	0.03518
9	58.247	226.	0.8	36.63083	0.65907	0.03553
10	58.532	221.	0.7	36.84989	0.65766	0.03574
11	62.121	6.	0.0	39.83444	0.63410	0.03428

STAGE	LKEY FRAC	HKEY FRAC	RATIO
1	0.99985	0.00015	6750.21240
2	0.99956	0.00044	2247.87305
3	0.99893	0.00107	931.95142
4	0.99762	0.00238	419.22925
5	0.99494	0.00506	196.54501
6	0.99494	0.00506	196.59381
7	0.99494	0.00506	196.46605
8	0.99487	0.00513	193.83403
9	0.99386	0.00614	161.97292
10	0.97960	0.02040	48.01899
11	0.80003	0.19997	4.00083

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